Fabrication of OH-free, single-mode fiber by using slurry casting and rod-in-tube method

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Abstract: We present a new simple and low-cost process for fabricating low-loss single-mode fiber (SMF) cladding preforms by a modified slurry casting method, in which the cladding preform and a core rod obtained separately with the VAD method are assembled by a rod-in-tube method. The fabricated SMF showed almost the same loss characteristics as a standard SMF with a minimum loss of 0.19 dB/km at 1550 nm and an OH-free loss spectrum, which were realized with an OH reduction process in a slurry casting based cladding. This OH reduction method plays an important role in preventing OH diffusion into the core and inner cladding.

Keywords: single-mode fiber, rod-in-tube method, slurry casting method, multi-core fiber

Classification: Fiber optics, Microwave photonics, Optical interconnection, Photonic signal processing, Photonic integration and systems

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1 Introduction

The data traffic in backbone optical networks has been increasing continuously due to the rapid growth of the Internet, mobile phones, and broadband services. Single-mode fibers (SMFs) have been supporting the large capacity of optical transport networks since the 1980's. Since the first demonstration of optical fiber with a transmission loss of 20 dB/km [1], a number of fabrication processes for low-loss optical fibers have been developed, including modified chemical vapor deposition (MCVD) [2], outside vapor deposition (OVD) [3], vapor-phase axial deposition (VAD) [4], and sol-gel methods [5]. The MCVD, OVD and VAD methods are widely used in current industrial processes. Because these processes are based on a chemical vapor deposition process with highly pure materials, very few impurities are included in the preform. On the other hand, with the sol-gel casting method, it is generally difficult to obtain a defect free gel body because voids and bubbles are inevitably generated in the gel body during the gelation process. Because of these issues, it has been difficult to reduce loss, especially the OH absorption loss. Moreover, with preform drilling it is difficult to maintain longitudinal structural uniformity.

Recently, an optical fiber preform fabrication process with a sand clad process was reported [6]. In this process, silica sand is filled by gravity between a core rod and a large thin wall tube. The sand is then fused into glass to fabricate a preform. This process can realize a cost effective optical fiber preform fabrication process. Other processes that use silica powder as a starting material were also proposed [7, 8]. One is a hybridized extrusion forming process [7], in which the core rod is surrounded with ductile pure silica soot (or clay), and the glass preform is obtained through degreasing, purification and VAD method. The other is a cold isostatic press (CIP) forming process [8], which consists of over-cladding of a VAD-derived core rod with commercial silica powder by CIP technique. However, optical fibers fabricated by silica sand or powder processes as described above [6, 7, 8] show relatively high transmission loss, imperfection loss and high OH absorption loss.

We recently proposed a novel process for fabricating a low-loss photonic crystal fiber (PCF) that employs a modified slurry casting method to make a pure silica PCF preform [9]. This process results in simple and low-cost PCF fabrication and provides improved precision and a highly flexible geometry in the air-hole structure. PCF fabricated by this process exhibits a minimum transmission loss of 1.1 dB/km at 1.55 μm. The result strongly suggests that silica glass fabricated by

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the slurry casting method should also be directly applicable to SMF cladding although this process is also one of the silica powder processes. We have also succeeded in fabrication of SMF using the slurry casting method [10].

In this paper, we describe the details of the SMF fabrication using the slurry casting method. Here the cladding preform made by the slurry casting method and a core rod obtained independently with the VAD method were assembled by a rod-in-tube method and drawn into fibers. The specification of the fabricated SMF is comparable to that of standard SMF fabricated by the VAD method, with a minimum loss of 0.19 dB/km at 1550 nm. The present preform fabrication technique offers the advantages of low cost, low complexity, and easy handling, and is very promising for application to multi-core fibers (MCFs).

2 Fabrication method

Fig. 1 shows the SMF preform fabrication process based on the slurry casting method. A highly pure SiO_2 powder, which is manufactured by the thermal oxidation of silicon chloride, is used as a starting material. The desired amount of SiO_2 powder, organic binder, dispersing agent and distilled water are mixed by ball milling for 16 hours. Polymerization initiator is added to the slurry, which is then poured into a metal mold. A stainless metal rod is positioned in the center of the mold to form a hole in the cladding preform for the insertion of a core rod. Once the organic binder has solidified by polymerization, the metal rod is removed and the preform is released from the mold. The obtained preform is dried carefully to prevent the generation of cracks. The dried preform is then calcined to remove organic chemicals by oxidation at a high temperature. The calcined preform is passed through a purification step that uses a chlorine gas to remove metallic impurities and OH from the preform, which involves sintering at 1400 °C. The fabricated silica glass cladding preform has an inner and outer diameter of 5.1 mm and 20 mm, respectively, with a length of 400 mm. The core rod, which is fabricated by the VAD method, has a diameter of 20 mm and a length of 800 mm, where a GeO_2-doped core (5 mm diameter) is surrounded by a SiO_2 cladding layer with a thickness of 7.5 mm. This core rod is drawn and the diameter is downsized to 5 mm in order to match the hole diameter of the cladding preform. The downsized core rod is cut to 400 mm length each. The core rod thus obtained is inserted in the hole of the sintered glass preform to form an SMF preform. Before constructing the SMF preform, the core rod and cladding preform are etched by 5%

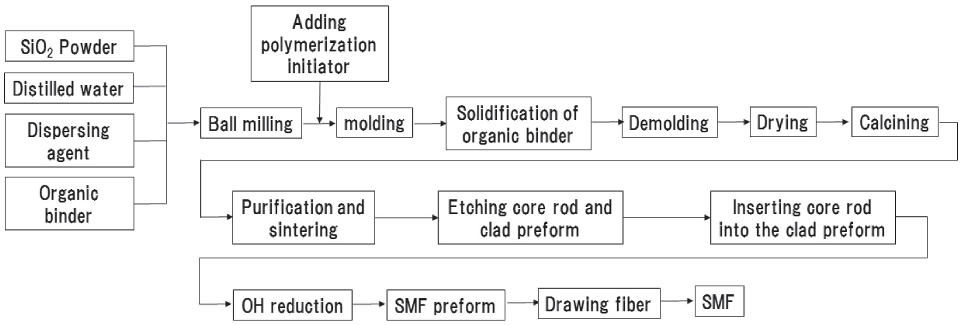

Fig. 1. SMF fabrication process based on slurry casting method.

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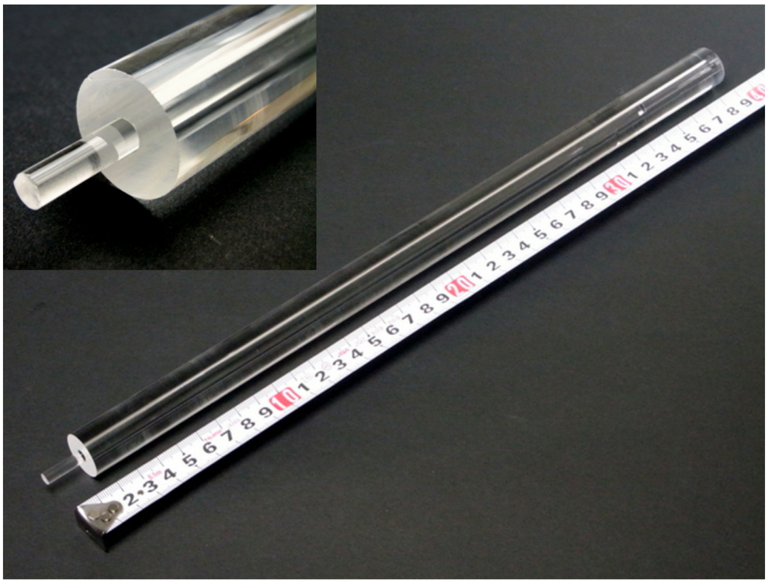

Fig. 2. Photograph of SMF preform fabricated by a slurry casting/rod-in-tube method.

HF solution for 4 hours at 23 °C to remove a silica glass imperfection layer and impurities on the surface of the core rod and cladding preform. An overview of the SMF preform constructed with the sintered cladding preform and the core rod is shown in Fig. 2. The constructed preform and dummy glass tube are spliced by heating with a H_2/O_2 burner. The preform thus obtained is treated in a chlorine gas containing helium atmosphere at 1200 °C for 0 to 60 minutes to remove OH from the surface of the cladding preform and the core rod. Several different conditions for preform treatment before drawing process are investigated, and the optimum chlorine concentration is determined for the present slurry casting/rod-in-tube method. The preform is finally drawn into a fiber at 2000 °C.

3 Optical properties of fabricated SMF

Fig. 3 shows the transmission loss of the fabricated SMF without a chlorine gas treatment before drawing. The transmission loss at 1.55 µm was 0.27 dB/km. The OH absorption peak was 0.44 dB/km at 1.38 µm. It has been reported that a PANDA fiber fabricated by the rod-in-tube method also exhibits a higher OH absorption loss than a conventional SMF, because the water molecules that remain on the surface of the hole in the preform can easily diffuse into the core area [11]. We used the coefficient of water diffusion into silica glass reported in Ref. [12] to estimate the water diffusion coefficient during our fiber drawing process. The water diffusion coefficient is estimated to be approximately 1–1.8 cm^2/sec in the 1800 to 2000 °C temperature range. During our drawing process, the fiber preform was heated for more than 30 minutes at 1800–2000 °C. According to Fick's diffusion law, the water diffusion distance is calculated to be 65–80 µm under this condition. The cladding thickness of the core rod used in this work was 1.75 mm and it decreased to 15 µm during the drawing process. This suggests that the water molecules on the surfaces of the core rod and the hole in the cladding preform can diffuse into the SMF core area during our drawing process.

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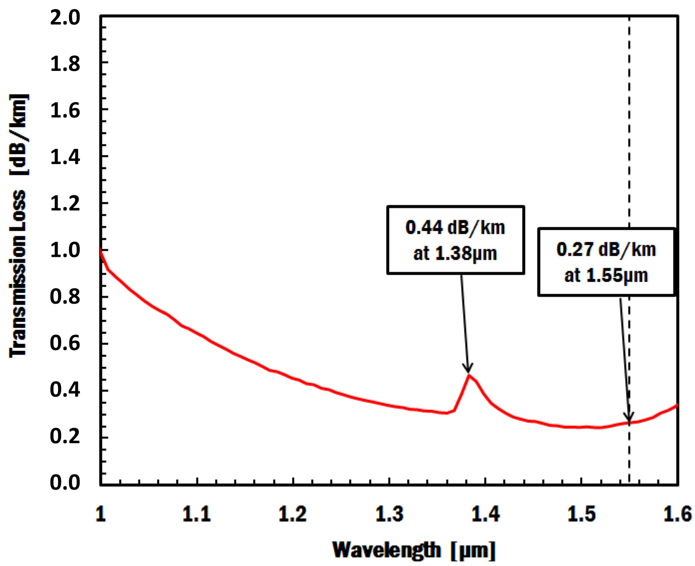

Fig. 3. Transmission loss of SMF fabricated by slurry casting and rod-in-tube method without chlorine gas treatment before drawing.

Table I. The chlorine gas treatment condition of SMF preforms fabricated by slurry casting and rod-in-tube method before drawing.

Sample No.	Cl_2 gas Conc.	Treatment temp.	Treatment time	Loss at 1.38 μm
	%	°C	min.	dB/km
1	0	1200	0	0.44
2	5	1200	60	0.38
3	20	1200	15	0.29
4	20	1200	30	0.29
5	20	1200	60	0.28
6	30	1200	60	0.26
Standard SMF	-	-	-	0.29

From the above estimation, the OH absorption peak is likely to be caused by water molecules adsorbed on the surface of the core rod and cladding preform during the rod-in-tube process because the core rod and the hole in the cladding preform are constructed in an ambient atmosphere containing water vapor. The adsorbed water molecules could easily dissolve and diffuse into the core rod and the cladding glass during drawing even if the core rod was manufactured by the VAD method.

As described above, the OH absorption loss at 1.38 μm is strongly influenced by the water molecules on the surfaces of the core rod and the hole in the cladding preform. To obtain OH-free SMF, the water molecules must be removed before the fiber drawing process. To reduce the OH absorption loss, we treated the constructed SMF preforms in the atmosphere containing 5 to 30% of chlorine gas and containing helium atmosphere at 1200 °C for 15 to 60 minutes before the drawing process. During the treatment with chlorine gas, most of the water molecules adsorbed on the surface of the core rod and cladding preform were removed. As a result, an OH-free loss spectrum was obtained.

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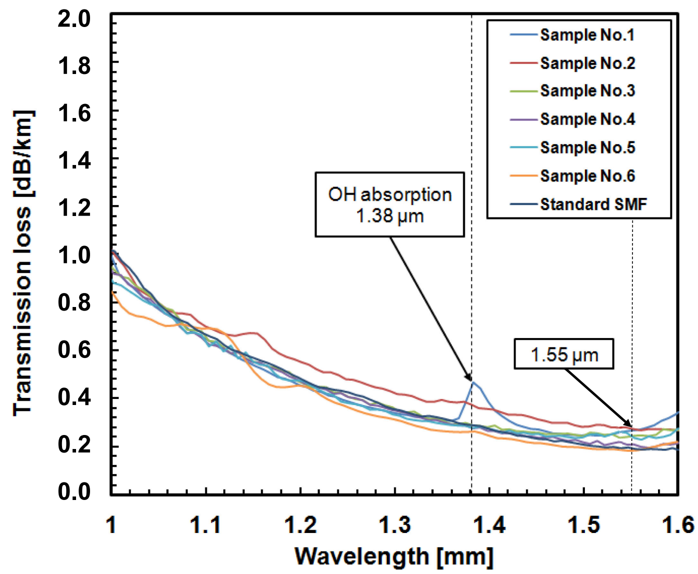

Fig. 4. Transmission loss of SMFs treated with various chlorine gas treatment conditions.

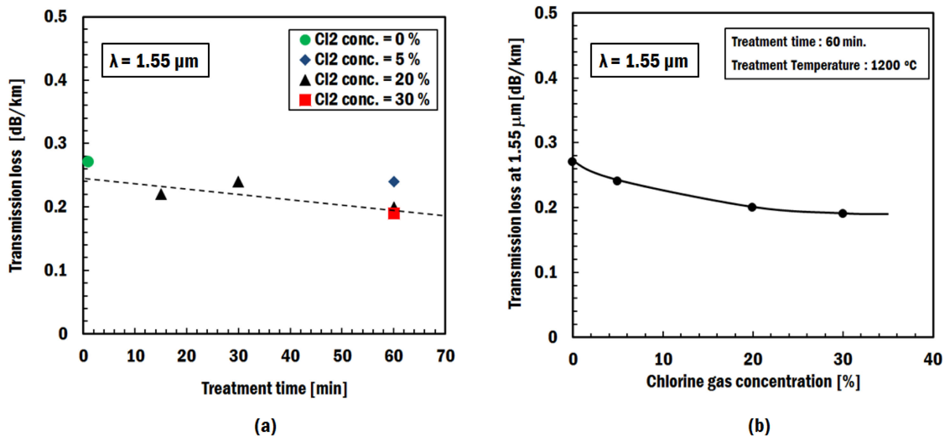

Fig. 5. The dependence of SMF transmission losses at 1.55 μm on chlorine gas treatment conditions. (a) Dependence on treatment time, (b) dependence on chlorine gas concentration.

The conditions of the chlorine gas treatments are summarized in Table I. OH absorption peak was successfully suppressed as a result of chlorine gas treatment as shown in Fig. 4. This is in contrast to SMFs fabricated by conventional hybrid fiber preform processes with extrusion and CIP forming [7, 8], which exhibit relatively high OH absorption loss.

Fig. 5 shows the SMF transmission loss at 1.55 μm under various conditions for chlorine gas treatment. Longer treatment time results in lower transmission losses as shown in Fig. 5(a). Furthermore, the transmission loss decreased with the increase of the chlorine gas concentration in the atmosphere as shown in Fig. 5(b). The minimum transmission loss of as low as 0.19 dB/km was obtained at 1.55 μm in the sample No. 6 which was treated with 30% chlorine gas containing atmosphere at 1200 °C for 60 minutes. These results indicate that the chlorine gas treatment is useful not only for reduction of OH absorption loss but also for total loss decrease.

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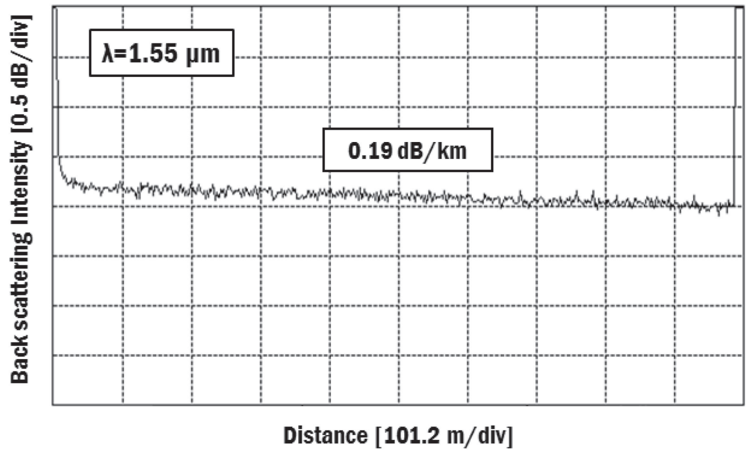

Fig. 6. OTDR waveform of SMF fabricated by slurry casting and rod-in-tube method. Fiber length is 1 km.

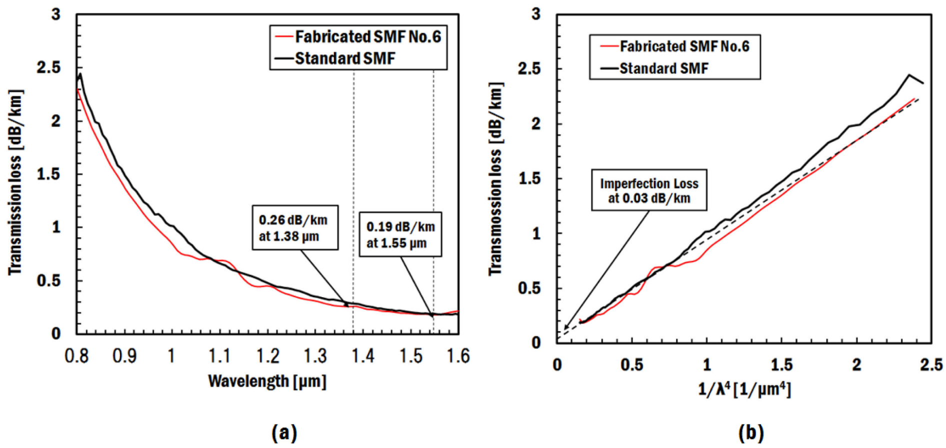

Fig. 7. Transmission loss of SMF fabricated by slurry casting method under the optimum chlorine gas treatment condition (a) and its λ^{-4} plot (b). Fiber length is 1 km. The red line shows the transmission loss of SMF fabricated by the slurry casting method. The black line shows that of a standard SMF.

Fig. 6 shows an optical time-domain reflectometer (OTDR) waveform of a 1 km SMF fabricated under the optimum chlorine gas treatment condition (sample No. 6), measured at 1.55 μm. As shown in Fig. 6, the SMF was uniform along its length, and no discontinuities were observed. This result suggests that the cladding preform fabricated by the slurry casting method functions successfully as an SMF cladding material and the fiber has longitudinal structural uniformity.

Fig. 7 shows the transmission loss spectrum of the fabricated SMF, and compares it with a standard SMF fabricated by the VAD method. The transmission loss of the obtained SMF is 0.19 dB/km at 1.55 μm and 0.26 dB/km at 1.38 μm, which are identical to a standard SMF. Under the present OH reduction process, an OH absorption-free loss spectrum was successfully obtained despite the use of cladding glass material fabricated by the slurry casting and rod-in-tube method.

The measured transmission loss was fitted to the expression,

$$\alpha = A/\lambda^4 + B + \alpha_{OH} + \alpha_{IR}, \tag{1}$$

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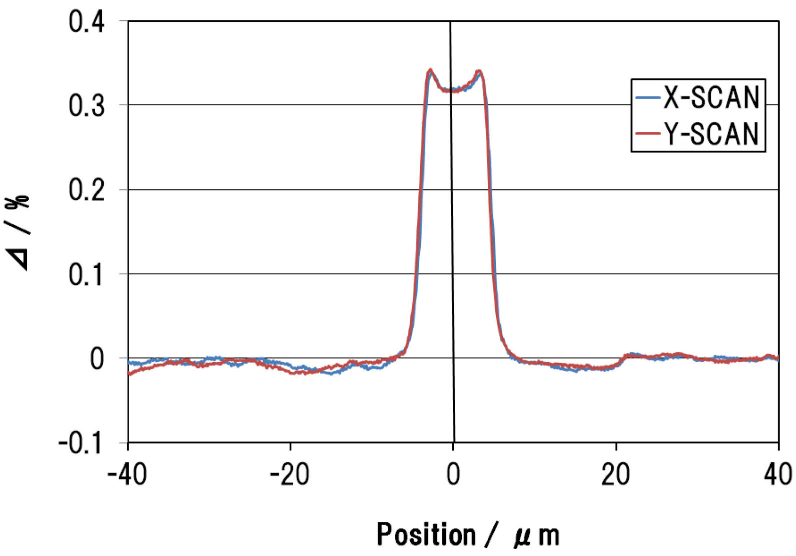

Fig. 8. Index profile of the fabricated SMF.

where α, λ, A, B, α_{OH} and α_{IR} are the transmission loss, wavelength, Rayleigh scattering coefficient, imperfection loss, OH absorption loss and infrared absorption loss, respectively. The λ^{-4} plot of the loss profile is shown in Fig. 7(b). From this plot, we estimated the Rayleigh scattering coefficient to be 0.92 dB/km·μm^4 and the imperfection loss to be as low as 0.03 dB/km, which indicates that there are very few structural imperfections between the core and the cladding. These values are comparable to those of a standard SMF.

Fig. 8 shows an index profile of the fabricated SMF. A trench-like slight distortion is observed in the cladding area at 5~20 µm from the center, where the outer edge of the trench corresponds to the boundary between the core rod and the fabricated cladding. It can be seen that the cladding index of the fabricated SMF is almost the same as that of the core rod, and it suggests that the cladding obtained by the present slurry casting method successfully functions as an SMF cladding material.

Fig. 9 shows the Weibull distribution of tensile strength of the fabricated SMF. No weak points are observed. This suggests that there are no micro crack and breaking point on the surface of the fabricated SMF.

The present process for fabricating a silica cladding preform can also be expected to be directly applicable to MCF. MCF preforms have been fabricated by a stack and draw method or a rod-in-tube method [13]. The stack and draw method involves complicated processes for manually assembling a number of glass rods with high precision. The rod-in-tube method also requires ultrasonic drilling and polishing of the inner surfaces of holes to remove surface imperfections. The cladding rod glass may be damaged during the drilling process, and in addition, there is a limitation to the preform size because of the size of the drilling tool. As reported in [9], the slurry casting method has already demonstrated the capability for fabricating many holes in silica cladding preform with high precision, and can be applicable to fabricate MCF preforms at a substantially lower cost, with higher flexibility in terms of multi-core geometry, and scalability as regards the number of cores.

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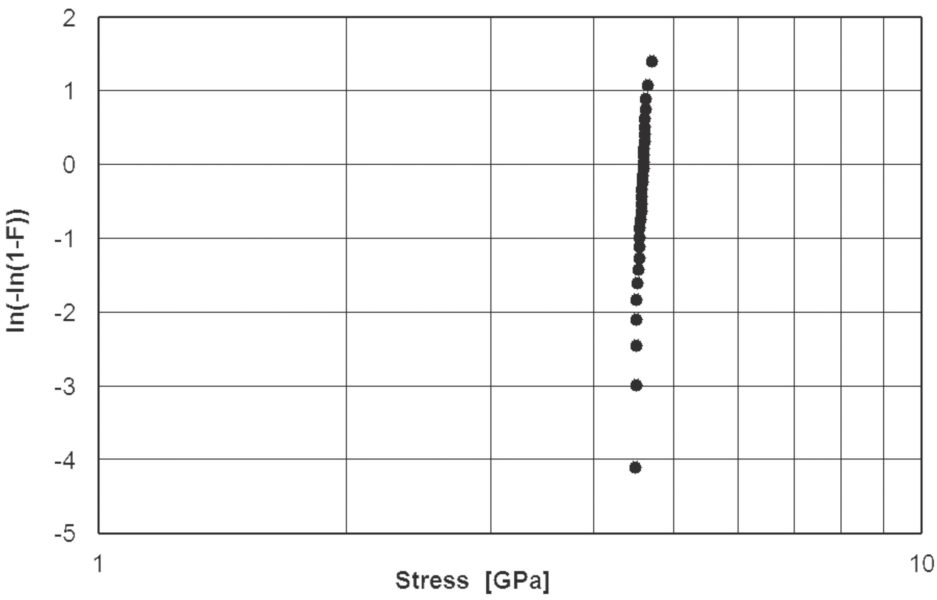

Fig. 9. Tensile strength of the fabricated SMF.

4 Conclusion

We proposed a novel technique for fabricating low-loss silica cladding preforms by a modified slurry casting method and the drawing of SMF with a rod-in-tube method. The SMF thus obtained showed almost the same loss characteristics as a standard SMF fabricated by the VAD method, with a minimum loss of 0.19 dB/km at 1550 nm and an OH-free loss spectrum. The OH reduction process of constructed preforms by chlorine gas treatment at high temperature is very effective the reduction of not only OH absorption at 1.38 µm but also the transmission loss at 1.55 µm. The slurry casting method is very convenient and attractive for fabricating SMF cladding preforms, and offers good potential for use in fabricating MCF cladding preforms at low cost and with a simple process.

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