

Description

Field of the invention

The present invention relates to a method of producing a glass preform for use in the fabrication of an optical fiber having good light transmission characteristics. More particularly, it relates to such a method wherein a core rod made of quartz glass is inserted into a cladding tube made of quartz glass added with fluorine. These are fused together by heating them from the outside of the cladding tube to form a primary glass preform comprising a core rod and a first cladding layer around the core rod.

Background of the invention

A single mode optical fiber comprising a core made of pure quartz glass and a cladding made of fluorine added quartz glass theoretically has small attenuation of light transmission since the core does not contain any additive which causes scattering loss. Since pure quartz glass has less structural defects, it has good resistance to irradiation, which varies with the amount of the defects. Therefore, once such optical fiber is fabricated, it can be used as a reliable optical fiber suitable for telecommunication with a large capacity. However, such optical fiber is difficult to produce. For example, when it is produced by a known modified chemical vapor deposition (MCVD) method, it is difficult to increase a synthesizing rate of the cladding made of the fluorine added quartz glass, which means poor productivity of the optical fiber. By a vapor phase axial deposition (VAD) method, it is difficult to form a refractive index distribution by the addition of fluorine.

As one of the methods for producing a glass preform for use in the fabrication of an optical fiber, a so-called rod-in-tube method is known. The rod-in-tube method comprises inserting, as a core material, a glass rod having a higher refractive index in a quartz glass tube having a lower refractive index than that of the core material and heating and fusing them together, namely collapsing them to form a glass preform. However, by the rod-in-tube method, it is difficult to reduce the attenuation of light transmission although it is easy to form a refractive index distribution.

As the results of the study by the present inventors, the following is found:

For the production of a glass preform from which a single mode optical fiber having a larger ratio of a cladding diameter to a core diameter is to be drawn, it is difficult to form a cladding layer with a desired thickness in a single step by the rod-in-tube method. That is, when the core rod has a small outer diameter, it is easily deformed in the collapsing step so that the core portion of the resulting glass preform tends to be eccentric. When the core rod has a large outer diameter, the wall thickness of the quartz tube should be made large. Therefore, it is rather difficult to collapse them.

It is proposed to collapse a plural of tubes made

of quartz glass added with fluorine successively around the core rod made of pure quartz glass. By this method, eccentricity of the core is suppressed or prevented and all the cladding layers can be formed with good thermal efficiency. When an optical fiber is fabricated from the glass preform produced by this method, absorption loss of the optical fiber due to, for example, residual hydroxyl groups can be decreased by suitable treatment of the preform before or during collapsing. However, it is found that the optical fiber still has some attenuation of light transmission which is independent on the wavelength. Further, in the above method, since several collapsing steps are required, a large amount of materials is wasted so that the productivity of the final optical fiber is not sufficient.

US-A-3 932 162 discloses forming a clad optical waveguide fiber by firstly forming an intermediate fiber by heating and then drawing a two-layer glass structure. The intermediate fiber is then placed within a hollow glass tube, which is then heated and drawn to reduce the cross-sectional area thereof to form the optical waveguide fiber.

Japanese unexamined application No. 57-170831 discloses the production of a fluorine containing porous base material from which an optical fiber may be manufactured. The base material is produced by heating a porous preform existing of a core and a clad out of fine oxide glass particles in a container into which a gas such as F_2 , HF, NH_4F or $NH_4F \cdot F$ is fed. This heating procedure converts the core and cladding into the fluorine containing porous base material.

WO-A-80 01378 discloses the production of an intermediate preform structure from which glass fiber optical transmission lines may be produced. The intermediate preform has a core-to-clad ratio greater than that desired in the ultimate fiber, and this is obtained by depositing the core by means of a MCVD process, prior to the completion of the cladding. Additional material may be added to complete the cladding subsequent to the deposition of the internal core thereby yielding an optical fiber preform having the desired core-to-clad ratio.

FR-A-2 441 594 discloses the manufacture of an optical fiber waveguide by inserting a silica glass rod into a silica glass tube, and then heating and drawing the structure to form the fiber. Simultaneously with the heating, the vapor of a hydrogen-free compound is passed between the tube and rod. The method defined in the pre-characterising part of claim 1 is known from this document.

Summary of the invention

One object of the present invention is to provide a method for producing a glass preform for use in the fabrication of a single mode optical fiber having good light transmission characteristics by a rod-in-tube method.

Another object of the present invention is to provide a method for producing a glass preform

for use in the fabrication of an optical fiber having low attenuation of light transmission in a wide range of wavelength.

A further object of the present invention is to provide a method for producing a glass preform for use in the fabrication of an optical fiber by which the cladding tube is easily collapsed so that eccentricity of the core rod is suppressed or prevented.

According to the present invention there is provided a method of producing a glass preform for use in the fabrication of an optical fiber, which comprises inserting a core rod made of quartz glass in a cladding tube made of quartz glass, heating them from the outside of the cladding tube to fuse them together to form a primary glass preform comprising a core rod and a first cladding layer around the core rod, characterized by the quartz glass of the cladding tube being added with fluorine and by depositing SiO_2 soot particles around the primary glass preform and thermally treating the soot deposited glass preform in the presence of a fluorine-containing compound to form a final glass preform from which an optical fiber can be fabricated.

Brief description of the drawings

Fig. 1 shows a relationship between the absorption peak at a wavelength of $1.38 \mu\text{m}$ and the cladding diameter/core diameter ratio of the primary glass preform, and

Fig. 2 shows attenuation characteristics of the optical fibers produced in Example 1 (Curve A), Example 2 (Curve B) and Comparative Example 1 (Curve C).

Detailed description of the invention

In the method of the present invention, firstly, the primary glass preform having a comparatively small ratio of the cladding diameter and the core diameter is produced by inserting the core rod in the glass tube made of quartz glass added with fluorine (hereinafter referred to as "glass tube") and collapsing the glass tube onto the core rod.

The core rod is preferably made of a highly pure glass material containing few or no residual hydroxyl group. Such core rod can be prepared by the conventional VAD method.

The glass tube is also preferably made of a highly pure glass material containing few or no residual hydroxyl group. Such glass tube can be prepared by forming a soot preform of pure SiO_2 particles by the conventional VAD method, dehydrating it in an atmosphere containing a chlorine-containing compound, adding fluorine to it with a fluorine-containing compound (e.g. SF_6 , CF_4 and SiF_4), vitrifying the soot preform to produce a transparent glass rod and boring it to form a glass tube.

The composite of the cladding glass tube and the core rod inserted in the tube is then heated and collapsed at a temperature higher than a flowing point of the glass, for example, a temperature of 1,400 to 1,800, preferably 1,500 to 1,700. In this step, preferably, the inner wall

surface of the glass tube and the outer surface of the glass rod are cleaned, for example, by gas phase etching with a halogen-containing compound (e.g. SiF_4 , Cl_2 , SF_6 , SOCl_2 , SiCl_4 and the like).

Preferably, the primary glass preform produced by the above steps has a first cladding diameter/core diameter ratio of 3 to 7. When the ratio is large, the interfacial condition between the first and second claddings has less influence on the attenuation characteristics of the fabricated optical fiber, but collapsing of the composite is difficult. Fig. 1 shows the relationship between absorption peak of the optical fiber at a wavelength of $1.38 \mu\text{m}$ and the ratio of the first cladding diameter and the core diameter.

In the next step, around the primary glass preform, soot particles of SiO_2 are deposited.

Usually, the outer diameter of the primary glass preform is 10 to 20 mm. When it is too small, the primary glass preform is deformed by shrinking force of the layer of the deposited SiO_2 particles during the subsequent heating step. When the outer diameter of the primary glass preform is too large, the layer of the deposited SiO_2 particles should be made large but the thicker layer tends to cause cracking of the layer. The SiO_2 particles are produced by flame oxidation of a glass-forming raw material (e.g. SiCl_4 , SiBr_4 , SiF_4 and the like) with, for example, oxyhydrogen flame or natural gas flame and deposited in a semi-sintered state on the surface of the primary glass preform.

In the subsequent heating step, the preform having the layer of the SiO_2 particles is heated in an atmosphere comprising the fluorine-containing compound (e.g. SF_6 , CF_4 and SiF_4) at a temperature of 1,000 to $1,700^\circ\text{C}$, preferably $1,300$ to $1,600^\circ\text{C}$. Heating of the glass preform may be carried out by gradually inserting it in a furnace kept at a desired elevated temperature or placing it in a furnace kept at a low temperature and gradually raising the temperature of the furnace. Fluorine is added in such an amount that the refractive indexes of the first and second cladding are substantially the same. For example, the difference of the refractive indexes between the first and second cladding is 0.05% or less. If the difference of the refractive indexes is larger than this limit, the fabricated optical fiber has some increased long wavelength loss or unstable cut off value.

Practical and presently preferred embodiments of the present invention are shown in the following examples.

Example 1

A highly pure quartz glass synthesized by the VAD method was drawn at $2,000^\circ\text{C}$ to form a rod having an outer diameter of 3 mm and a length of 50 cm.

A pure SiO_2 glass soot rod was produced by the VAD method and then heated by gradually introducing it in a furnace in three steps under following conditions to produce a fluorine-added glass

rod of 50 mm in outer diameter and 300 mm in length having a refractive index 3% lower than that of quartz glass:

	Temperature	Atmosphere
Step 1:	1,000	Cl ₂ 3%, He 97%
Step 2:	1,350	SiF ₄ 3%, He 97%
Step 3:	1,650	SiF ₄ 3%, He 97%

Then, the rod was bored at its center and drawn to form a fluorine-added quartz tube of 20 mm in outer diameter and 4 mm in inner diameter.

The pure quartz rod was inserted in the fluorine-added quartz glass tube and then heated and collapsed with introducing a gaseous mixture of SiF₄, Cl₂ and oxygen (ratio=1:1:6 by volume) through a gap between the rod and the tube to form the primary glass preform having an outer diameter of 19 mm and a first cladding diameter/core diameter ratio of 6.6. The primary preform was drawn to an outer diameter of 14 mm.

On the outer surface of the drawn primary preform, SiO₂ soot particles were deposited to an outer diameter of 120 mm. The soot particles were synthesized by introducing SiCl₄ in an oxyhydrogen flame. The preform having the deposited particles was placed in a furnace kept at 800°C and heated to 1,500°C at a rate of 3.3°C/min with introducing helium gas and SiF₄ gas at rates of 15 l/min and 450 ml/min, respectively.

The produced glass preform was drawn to fabricate a single mode optical fiber having an outer diameter of 125 μm. The optical fiber had a core diameter of 8.3 μm, and a difference of specific refractive indexes of the cladding and the core of 0.3%. Its attenuation of light transmission at a wavelength of 1.3 μm was 0.35 dB/km and absorption peak at a wavelength of 1.38 μm due to the hydroxyl groups was 1.5 dB/km.

Its attenuation characteristics is shown in Fig. 2, Curve A.

Example 2

In the same manner as in Example 1 except that the first cladding diameter/core diameter ratio was 3, a single mode optical fiber was fabricated. Its attenuation of light transmission at a wavelength of 1.3 and 1.38 μm was 0.45 and 7 dB/km, respectively. Its attenuation characteristics is shown in Fig. 2, Curve B.

Comparative Example 1

The primary glass preform produced in the same manner as in Example 1 was covered with a glass tube made of a quartz glass added with fluorine and collapsed to form a second cladding. From the thus produced preform, a single mode optical fiber having substantially the same cross sectional structure as that of the optical fiber fabricated in Example 1 was fabricated. Its attenuation of light transmission at a wavelength of 1.3 and 1.38 μm was 0.52 and 1.6 dB/km, respectively. Although it contained less residual hydroxyl

groups, attenuation was increased by about 0.1 dB/km or more in a wide range of wavelength. Its attenuation characteristics is shown in Fig. 2, Curve C.

Comparative Example 2

Around the same glass rod having an outer diameter of 7 mm produced in the same manner as in Example 1, soot particles of SiO₂ was deposited in the same manner as in Example 1 to an outer diameter of 80 mm. Then, the glass rod having the layer of the deposited soot particles was heated in an atmosphere comprising helium gas and a fluorine-containing compound (SiF₄) to form a primary glass preform having a cladding diameter/core diameter ratio of 5.

The primary preform was drawn to an outer diameter of 7 mm and, then, soot particles of SiO₂ were deposited around it to form a second cladding in the same manner as in Example 1. From the preform, an optical fiber having substantially the same cross sectional structure as that of the optical fiber as fabricated in Example 1 was fabricated. Its attenuation of light transmission at a wavelength of 1.3 and 1.38 μm was 1.4 and 30 dB/km, respectively. This means that the optical fiber had large absorption peak due to the hydroxyl group, which might be formed by the contact of the oxyhydrogen flame with the core rod.

From the glass preform produced by the method of the present invention, an optical fiber having extremely low attenuation of light transmission in a wide range of wavelength is fabricated.

Since the first cladding can be made thin, heat efficiency in the collapsing step is improved.

As the primary glass preform is used as a rod member on which the soot particles of the second cladding is deposited, any glass tube previously synthesized is not used as the second cladding. Therefore, such processes as cleaning of the inner surface of the glass tube or collapsing of the tube is not necessary, this resulting in high productivity of the glass preform.

Since the second cladding can be formed after the primary glass preform is drawn, the primary glass preform may have a larger size and a smaller cladding diameter/core diameter ratio so that the collapsing of the cladding is easily carried out, and eccentricity of the core is prevented. Therefore, the fabrication of inferior optical fibers is suppressed and, in turn, the productivity of the optical fiber is increased.

Claims

1. A method of producing a glass preform for use in the fabrication of an optical fiber, which comprises inserting a core rod made of quartz glass in a cladding tube made of quartz glass, heating them from the outside of the cladding tube to fuse them together to form a primary glass preform comprising a core rod and a first cladding layer around the core rod, characterized



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rod of 50 mm in outer diameter and 300 mm in length having a refractive index 3% lower than that of quartz glass:

	Temperature	Atmosphere
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Then, the rod was bored at its center and drawn to form a fluorine-added quartz tube of 20 mm in outer diameter and 4 mm in inner diameter.

The pure quartz rod was inserted in the fluorine-added quartz glass tube and then heated and collapsed with introducing a gaseous mixture of SiF₄, Cl₂ and oxygen (ratio=1:1:6 by volume) through a gap between the rod and the tube to form the primary glass preform having an outer diameter of 19 mm and a first cladding diameter/core diameter ratio of 6.6. The primary preform was drawn to an outer diameter of 14 mm.

On the outer surface of the drawn primary preform, SiO₂ soot particles were deposited to an outer diameter of 120 mm. The soot particles were synthesized by introducing SiCl₄ in an oxyhydrogen flame. The preform having the deposited particles was placed in a furnace kept at 800°C and heated to 1,500°C at a rate of 3.3°C/min with introducing helium gas and SiF₄ gas at rates of 15 l/min and 450 ml/min, respectively.

The produced glass preform was drawn to fabricate a single mode optical fiber having an outer diameter of 125 μm. The optical fiber had a core diameter of 8.3 μm, and a difference of specific refractive indexes of the cladding and the core of 0.3%. Its attenuation of light transmission at a wavelength of 1.3 μm was 0.35 dB/km and absorption peak at a wavelength of 1.38 μm due to the hydroxyl groups was 1.5 dB/km.

Its attenuation characteristics is shown in Fig. 2, Curve A.

Example 2

In the same manner as in Example 1 except that the first cladding diameter/core diameter ratio was 3, a single mode optical fiber was fabricated. Its attenuation of light transmission at a wavelength of 1.3 and 1.38 μm was 0.45 and 7 dB/km, respectively. Its attenuation characteristics is shown in Fig. 2, Curve B.

Comparative Example 1

The primary glass preform produced in the same manner as in Example 1 was covered with a glass tube made of a quartz glass added with fluorine and collapsed to form a second cladding. From the thus produced preform, a single mode optical fiber having substantially the same cross sectional structure as that of the optical fiber fabricated in Example 1 was fabricated. Its attenuation of light transmission at a wavelength of 1.3 and 1.38 μm was 0.52 and 1.6 dB/km, respectively. Although it contained less residual hydroxyl

groups, attenuation was increased by about 0.1 dB/km or more in a wide range of wavelength. Its attenuation characteristics is shown in Fig. 2, Curve C.

Comparative Example 2

Around the same glass rod having an outer diameter of 7 mm produced in the same manner as in Example 1, soot particles of SiO₂ was deposited in the same manner as in Example 1 to an outer diameter of 80 mm. Then, the glass rod having the layer of the deposited soot particles was heated in an atmosphere comprising helium gas and a fluorine-containing compound (SiF₄) to form a primary glass preform having a cladding diameter/core diameter ratio of 5.

The primary preform was drawn to an outer diameter of 7 mm and, then, soot particles of SiO₂ were deposited around it to form a second cladding in the same manner as in Example 1. From the preform, an optical fiber having substantially the same cross sectional structure as that of the optical fiber as fabricated in Example 1 was fabricated. Its attenuation of light transmission at a wavelength of 1.3 and 1.38 μm was 1.4 and 30 dB/km, respectively. This means that the optical fiber had large absorption peak due to the hydroxyl group, which might be formed by the contact of the oxyhydrogen flame with the core rod.

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Claims

1. A method of producing a glass preform for use in the fabrication of an optical fiber, which comprises inserting a core rod made of quartz glass in a cladding tube made of quartz glass, heating them from the outside of the cladding tube to fuse them together to form a primary glass preform comprising a core rod and a first cladding layer around the core rod, characterized

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by the quartz glass of the cladding tube being added with fluorine and by depositing SiO_2 soot particles around the primary glass preform and thermally treating the soot deposited glass preform in the presence of a fluorine-containing compound to form a final glass preform from which an optical fiber can be fabricated.

2. A method according to claim 1, further comprising the step of manufacturing the core rod by the VAD method.

3. The method according to claim 1, wherein, during collapsing, the inner surface of the cladding tube and the outer surface of the core rod are being gas phase etched.

4. The method according to claim 1, wherein the fused core rod and cladding tube are drawn before the SiO_2 soot particles are deposited.

5. The method according to claim 1, wherein the SiO_2 particles are synthesized by flame hydrolysis of SiCl_4 .

6. The method according to claim 1, wherein the thermal treatment is carried out at a temperature of 1,000 to 1,700°C.

Patentansprüche

1. Verfahren zur Herstellung einer Glasvorform für die Herstellung einer optischen Faser, bei dem ein Kernstab aus Quarzglas in ein Hüllrohr aus Quarzglas eingefügt wird, diese von der Aussen-seite des Hüllrohres aus erhitzt werden und unter Bildung einer primären Glasvorform aus einem Kernstab und einer ersten Hüllschicht um den Kernstab herum zusammenschmelzen, dadurch gekennzeichnet, dass dem Quarzglas des Hüllrohres Fluor beigefügt wird, und dass feine SiO_2 -Teilchen um die primäre Glasvorform herum abgeschieden werden, und die Glasvorform, auf der die feinen Teilchen abgeschieden wurden, in Gegenwart einer Fluor beinhaltenden Verbindung thermisch behandelt wird, wobei sich eine fertige Glasvorform bildet, aus der eine optische Faser hergestellt werden kann.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass es des weiteren die Herstellung des Kernstabes über das VAD-Verfahren umfasst.

3. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass während des Kollabierens die innere Oberfläche des Hüllrohres und die äussere Oberfläche des Kernstabes gasphasengeätzt werden.

4. Verfahren nach Anspruch 1, dadurch gekennzeichnet,

zeichnet, dass der geschmolzene Kernstab und das Hüllrohr gezogen werden, bevor die feinen SiO_2 -Teilchen abgeschieden werden.

5. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass die SiO_2 -Teilchen durch eine Flammenhydrolyse von SiCl_4 hergestellt werden.

6. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass die thermische Behandlung bei einer Temperatur von 1000 bis 1700°C durchgeführt wird.

Revendications

1. Un procédé de production d'une préforme en verre à utiliser dans la fabrication d'une fibre optique, ledit procédé comprenant l'insertion d'un coeur en forme de baguette réalisé en verre de quartz dans un tube de gainage réalisé en verre de quartz, le chauffage de ces éléments depuis l'extérieur du tube de gainage pour les fusionner ensemble et former une préforme primaire en verre comprenant un coeur en forme de baguette et une première couche de gainage autour du coeur en forme de baguette, caractérisé en ce que le verre de quartz du tube de gainage est additionné de fluor et en ce que l'on dépose des particules de suie de SiO_2 autour de la préforme primaire en verre et l'on traite thermiquement la préforme en verre avec le dépôt de suie en présence d'un composé contenant du fluor pour former une préforme finale en verre à partir de laquelle on peut fabriquer une fibre optique.

2. Un procédé selon la revendication 1, comprenant en outre l'étape de fabrication du coeur en forme de baguette par la méthode VAD.

3. Le procédé selon la revendication 1 selon lequel, durant l'effondrement, la surface interne du tube de gainage et la surface externe du coeur en forme de baguette sont soumises à une gravure en phase gazeuse.

4. Le procédé selon la revendication 1 selon lequel le coeur en forme de baguette et le tube de gainage fusionnés sont étirés avant le dépôt de particules de suie de SiO_2 .

5. Le procédé selon la revendication 1 selon lequel les particules de SiO_2 sont synthétisées par hydrolyse à la flamme du SiCl_4 .

6. Le procédé selon la revendication 1 selon lequel le traitement thermique est conduit à une température de 1 000 à 1 700°C.