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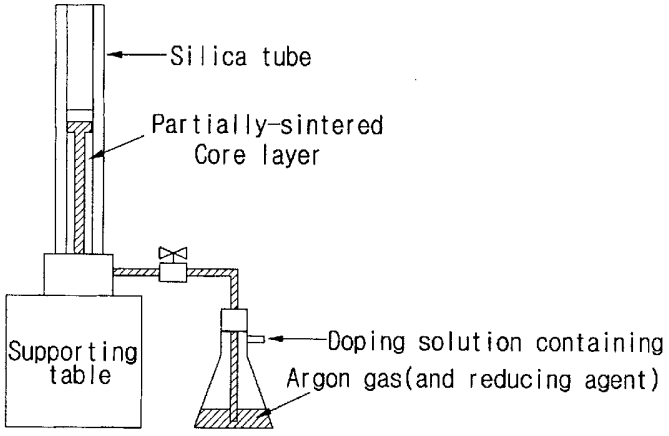
(56) Documents Cited:
EP 0517955 A1 **US 4610708 A**
WPI Abstract Accession No. 1991-243530 & JP 3159924 (Fujikura Ltd) 09/07/1991 (see abstract)
WPI Abstract Accession No. 1991-019603 & JP 2293332 (Shinetsu Chemical et al.) 04/12/1990 (see abstract)

(58) Field of Search:
UK CL (Edition X) **C1M**
INT CL[7] **C03B, C03C**
Other: **WPI and EPODOC**

(54) Abstract Title: **Doping an optical fibre preform with reduced metal ions**

(57) A method of fabricating an optical fibre or an optical device doped with reduced metal ion, in particular reduced rare earth metal ions, comprising the steps of: forming a partially sintered fine structure in a base material for fabricating the optical fibre or the optical device; soaking the fine structure in a doping solution containing a reducing agent together with a metal ion; then drying and sintering the fine structure. The reducing agent may be glucose, sucrose, glycerine, dextrin, benzene, phenol, hexane, toluene, naphthalene, or an alkoxide such as an tetraethyl orthosilicate or tetramethyl orthocarbonate. The valency of the metal ion is reduced, e.g. from Tm^{3+} to Tm^{2+}, thus changing the spectroscopic characteristics of the glass. The doped optical device may be used in optical switching and optical amplification applications.

FIG. 1



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United Kingdom

FIG. 1

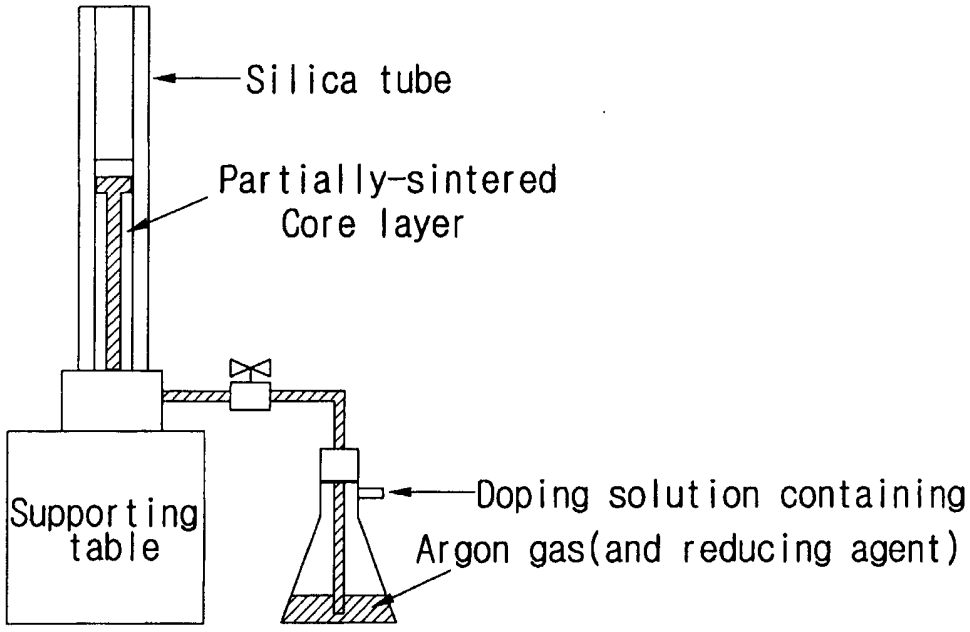

Silica tube
Partially-sintered
Core layer
Supporting
table
Doping solution containing
Argon gas(and reducing agent)

FIG.2

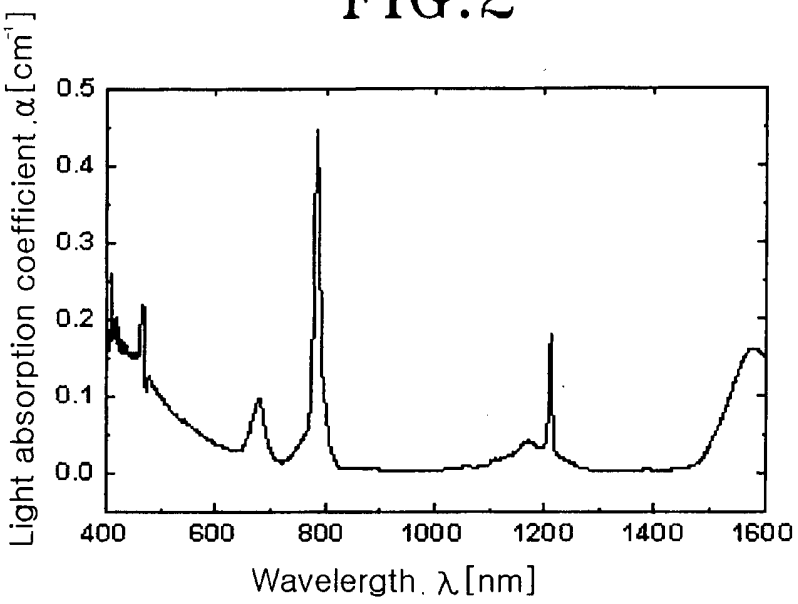

Light absorption coefficient, α[cm⁻¹]
0.5
0.4
0.3
0.2
0.1
0.0
400
600
800
1000
1200
1400
1600
Wavelergth, λ[nm]

FIG.3

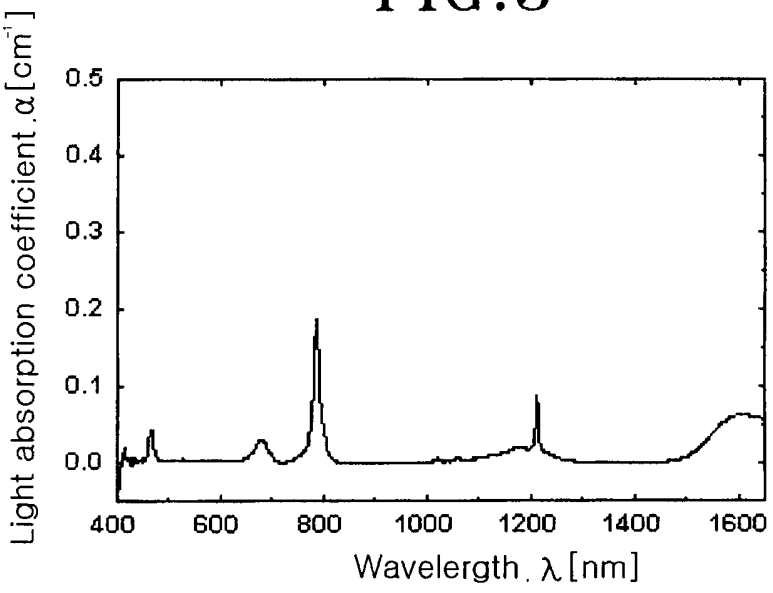

Light absorption coefficient, α[cm⁻¹]
0.5
0.4
0.3
0.2
0.1
0.0
400
600
800
1000
1200
1400
1600
Wavelergth, λ[nm]

FIG.4

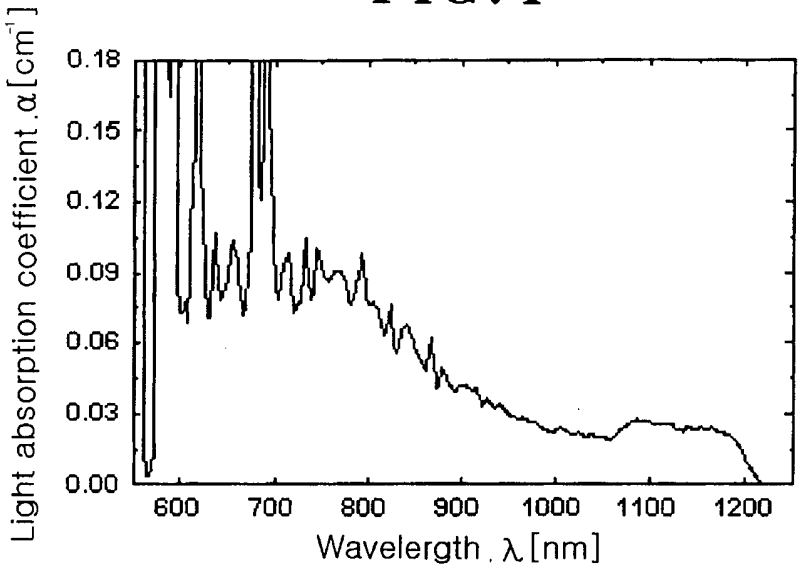
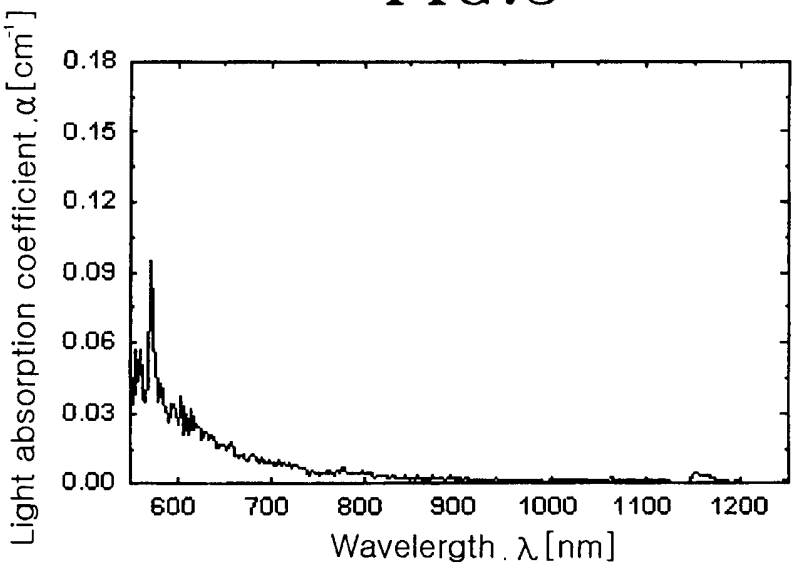

Light absorption coefficient, α[cm⁻¹]
0.18
0.15
0.12
0.09
0.06
0.03
0.00
600
700
800
900
1000
1100
1200
Wavelergth, λ[nm]

FIG.5


Light absorption coefficient, α[cm⁻¹]
0.18
0.15
0.12
0.09
0.06
0.03
0.00
600
700
800
900
1000
1100
1200
Wavelergth, λ[nm]

METHOD OF FABRICATING OPTICAL FIBER OR OPTICAL DEVICE DOPED WITH REDUCED METAL ION AND/OR RARE EARTH ION

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates generally to a technology for fabricating an optical fiber or an optical device, and more particularly to a method of fabricating an optical fiber or an optical device doped with reduced metal ion(s) and/or rare earth ion(s).

Description of the Related Art

An optical fiber containing metal ion and/or rare earth ion is brought under a special optical fiber, since it can be variously applied to an optical amplifier or an optical switching device etc. Therefore, much of the research in this area has been performed.

One of the research projects is a technique of reducing doped metal ion and/or rare earth ion. Generally, an atom has a different energy level distribution depending on its valence, and therefore has different spectroscopic characteristics such as light absorption and light emission. Accordingly, a little more diverse light absorption and light emission can be obtained by utilizing the change of valence and thereby the optical fiber and the optical device having various optical amplification and optical switching characteristics can be obtained.

As an example, let us consider rare earth ions, when a rare earth ion has the valence of 3+, the light absorption characteristic due to electronic transition

between $4f$ electron orbit and $5d$ electron orbit occurs only in an ultraviolet wavelength region, whereas when the valence of the rare earth ion changes to 2+ ion, such an light absorption characteristic occurs in both visible and infrared wavelength regions. For this reason, a technique of making doped metal ion and rare earth ion with desired valences, respectively is required. Furthermore, every atom has its own valence states in which the atom is mainly existed in nature and thus a specific process is required in order to transfer the valence into another valences.

For example, most of the rare earth ions have the valence of 3+. In order to stably transfer the valence of 3+ into the valence of 2+, 1+ or 0, it is necessary to reduce the rare earth ions. There have been proposed various reduction treatment methods as described below.

Firstly, there is a method of applying gamma rays to the rare earth metal ion having the valence of 3+. For example, it is reported that Tm^{2+} can be obtained, if the gamma rays is applied to a CaF_2 crystal containing Tm^{3+}.

However, in this method, there is a problem that a gamma ray source is dangerous to handle and the cost required in handling it safely is thus expensive.

Secondly, there is another method in which an aerosol type material is utilized. In this method, a MCVD (modified chemical vapor deposition) process is indispensable. In other words, this method includes the MCVD process in which a glass layer containing rare earth ions is deposited in a quartz glass tube, using material having aerosol formulation which generates carbon, together with a powder which generates rare earth ion and glass when fired. Then, processes of removing the carbon and OH radical, sintering the glass and collapsing the glass

tube are, in turn, performed to thus obtain an optical fiber preform. For example, in a glass optical fiber having SiO_2-Al_2O_3 components, Eu^{2+} and Sm^{2+} are reduced from Eu^{3+} and Sm^{3+}, respectively.

To date, this method which utilizes the material having aerosol formulation is performed through only the MCVD process. A desired rare earth ion material having aerosol formulation and an additional apparatus for supplying material having aerosol formulation are needed.

Further, there is a method of injecting a mixture of H_2 and Ar gases and obtaining the reduced rare earth ion during melting of glass. For example, in a glass having SiO_2-Al_2O_3 components or SiO_2-B_2O_3 components, Sm^{2+} is reduced from Sm^{3+}.

In this method, there is a problem that processes of fabricating the optical fiber preform are complicated in comparison with the conventional processes and are not yet commercialized.

SUMMARY OF THE INVENTION

Therefore, it is an object of the present invention to provide a method of fabricating an optical fiber or an optical device, in which metal ion and/or rare earth ion safely and facilely reduced, in comparison with the prior art methods, together with the utilization of the prior art processes of fabricating the optical fiber and/or the optical device.

To achieve the aforementioned object of the present invention, a method according to the present invention is characterized by forming a partially-sintered fine structure in a base material for fabricating an optical fiber or an optical device and soaking the fine structure into a doping solution containing a reducing agent

together with metal ion and/or rare earth ion during a selected time, thus doping the fine structure with the metal ion and/or rare earth ion together with the reducing agent. Therefore, reduced metal ion and/or rare earth ion through the reducing agent is obtained.

One method according to the present invention of fabricating an optical fiber or an optical device doped with reduced metal ion and/or rare earth ion comprising the steps of: forming a partially-sintered fine structure in a base material for fabricating the optical fiber or the optical device; soaking the fine structure into a doping solution containing a reducing agent together with metal ion and rare earth ion during a selected time; drying the fine structure in which the metal ion and rare ion is soaked; and heating the fine structure such that the fine structure is sintered.

Another method of fabricating an optical fiber or an optical device doped with reduced metal particle and/or rare earth element, comprising steps of: forming a partially-sintered fine structure in a base material for fabricating the optical fiber or the optical device; soaking the fine structure into a doping solution containing a reducing agent having strong reduction potential together with metal ion and rare earth ion during a selected time; drying the fine structure in which the metal ion and/or the rare earth ion is/are soaked; and heating the fine structure such that the fine structure is sintered, thereby forming the metal particle and/or the rare earth elements.

Preferably, the reducing agent is hydrocarbon compounds. Glucose, sucrose, glycerine, dextrin, benzene, phenol, hexane, toluene, stylene, naphthalene, and the like are exemplified.

In addition, the reducing agent is alkoxide compounds. TEOS(tetraethyl orthosilicate), TMOS(tetramethyl orthosilicate), TEOC(tetraethyl orthocarbonate), TMOC(tetramethyl orthocarbonate) and the like are exemplified.

Preferably, the metal ion and/or rare earth ion is at least one ion selected from the group consisting of Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Al, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Y, Zr, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, In, Sn, Hf, Ta, W, Re, Os, Ir, Pt, Au, Tl, Pb, Bi and a mixture thereof.

Further, the base material for fabricating the optical fiber or the optical device has a basic composition comprising a silicon oxide or a composite oxide of a silicone oxide and an oxide; in which the oxide is at least one selected from the group consisting of germanium oxide (GeO_2), boron oxide (B_2O_3), phosphorous oxide (P_2O_5), and titanium oxide (TiO_2).

Preferably, the base material for fabricating the optical fiber or the optical device has a basic composition selected from silica (SiO_2), germanosilicate (SiO_2-GeO_2), phosphorosilicate (SiO_2-P_2O_5), phosphorogermanosilicate (SiO_2-GeO_2-P_2O_5), borosilicate (SiO_2-B_2O_3), borophosphorosilicate (SiO_2-P_2O_5-B_2O_3), borogermanosilicate (SiO_2-GeO_2-B_2O_3), titanosilicate (SiO_2-TiO_2), phosphorotitanosilicate (SiO_2-TiO_2-P_2O_5), or borotitanosilicate (SiO_2-TiO_2-B_2O_3).

Preferably, the step of forming the partially-sintered fine structure in the base material for fabricating the optical fiber or the optical device is performed by a process selected from MCVD (modified chemical vapor deposition), VAD (vapor-phase axial deposition), VOD (outside vapor deposition), FHD (flame hydrolysis deposition), etc.

The optical device in the present invention includes a planar optical

amplifier, an optical communication laser, and a planar optical switch device, and the like.

BRIEF DESCRIPTION OF THE DRAWINGS

The above objects and another advantages of the present invention will become more apparent by describing in detail preferred embodiments thereof with reference to the attached drawings in which:

Fig. 1 is a schematic view showing an apparatus for performing processes of the present invention;

Fig. 2 is graph showing a light absorption spectrum of an optical fiber doped with reduced rare earth ion(Tm+2) fabricated by a first embodiment of the present invention;

Fig. 3 is a graph showing a light absorption spectrum of an optical fiber fabricated by a comparative example 1 without using the reducing agent;

Fig. 4 is a graph showing a light absorption spectrum of an optical fiber doped with reduced rare earth ion(Eu+2) fabricated by a second embodiment of the present invention;

Fig. 5 is a graph showing a light absorption spectrum of an optical fiber fabricated by a comparative example 2 without using the reducing agent.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Now, preferred embodiments of the present invention will be described in detail with reference to the annexed drawings.

A method of the present invention comprises a step of forming a partially-sintered fine structure in a base material for fabricating an optical fiber or an optical device, and a step of soaking the fine structure in a doping solution

containing a hydrocarbon compound as a reducing agent together with metal ion and/or rare earth ion for 1 to 1.5 hours. That is, the fine structure of the base material is doped with the reducing agent together with metal ion and/or rare earth ion and the metal ion and/or rare earth ion reduced by the doped reducing agent is thereby obtained.

The method of the present invention is a modification of a solution doping technique for adding the rare earth ion and/or metal ion to the optical fiber or the optical device. This solution doping technique is a method of doping the metal ion or the rare earth ion in an optical fiber core, which can be utilized with any of conventional methods of fabricating an optical fiber preform, such as MCVD (modified chemical vapor deposition), VAD (vapor-phase axial deposition), OVD (outside vapor deposition), etc. The solution doping technique is also used as the technique which may dope all of the rare earth ion and/or the metal ion, capable of being formed into a solution type, even in a method of fabricating a plane glass optical device through a FHD (flame hydrolysis deposition) process.

For example, the solution doping technique (please refer to J.E. Townsend, et al. "Solution for fabrication of rare earth doped optical fibers", Electron. Lett., Vol. 23, p.p. 329-331, 1987) through the MCVD process is as follows. Herein, in order to obtain reduced rare earth ion, an aqueous solution in which sucrose as a strong reducing agent is dissolved together with rare earth chloride is used as a doping solution. First, a core layer partially sintered and then having a plurality of pores is formed in a silica tube using the conventional MCVD process (please refer to MacChesney et. al., "Optical fiber fabrication and resulting product", U.S. Patent, 1997). Then, the silica tube is filled with the aqueous solution in which the

sucrose is dissolved together with the rare earth chloride. The aqueous solution is hold for 1 to 1.5 hours in order for the solution to be sufficiently permeated into the pores of the core layer and is then discharged. As a result, the doping solution remains in the pores. The core layer doped with the aqueous solution is dried while the silica tube is hold at a temperature of 100 to 250°C with the passage of an inert gas such as helium gas only, using the MCVD process. At this time, ethanol and moisture is removed. Sequentially, using hydrogen-oxygen flames, the core layer is heated at a high temperature of 2000°C until carbon generated from the sucrose is removed and the core layer is then completely sintered (referring to M.F. Yan, et al., "Sintering of optical wave-guide glasses", J. of Mater. Sci., p.p. 1371-1378, 1980). After that, the optical fiber preform is fabricated through a collapsing step in which the tube is heated to more than 2200°C with the continuous purging of the inert gas, using the hydrogen-oxygen flames. The optical fiber preform is drawn to produce the optical fiber doped with the reduced rare earth ion.

The sucrose contained in the doping solution is composed of C, H and O components. During the above drying step, most of the H and O components among the above components are removed and only the carbon (C) is remained. The carbon (C) is combined with Oxygen (O_2) remained at the high temperature of about 2000°C to form carbon monoxide (CO), and thus carbon monoxide reduces the doped rare earth ion. At this time, the reaction temperature at which the carbon monoxide (CO) is formed is decided within the possible range of reduction of the rare earth ion, using an Ellingham Diagram. At the same time, a strong reduction atmosphere is created by injecting only the inert gas into the silica glass tube, so that the carbon (C) can be fully participated in the reduction reaction of rare earth

ion. Further, preferably, the inert gas only is also passed through during the collapsing step for fabricating the optical fiber preform, thereby creating a reduction atmosphere at its maximum.

Embodiment 1

First, thulium chloride hexahydrate ($TmCl_3 \cdot 6H_2O$) of 0.04M and sucrose ($C_{12}H_{22}O_{11}$) of 2.17M are dissolved in deionized water to prepare a doping solution containing rare earth ion (Tm^{3+}) and the sucrose as a reducing agent. Herein, a hydrocarbon compound or an alkoxide compound is used as the reducing agent.

As shown in Fig. 1, a porous fine structure is formed through an MCVD process at an inner wall of a silica glass tube having an inner diameter of 19mm and an outer diameter of 25mm so that the portion thereof to form an optical fiber core has a basic glass composition of SiO_2-GeO_2. The doping solution fabricated is injected into the above glass tube and then discharged after 1 hour. Then, the core layer is dried by heating the glass tube again at a temperature of 100 to 250°C using the MCVD apparatus with the purge of only helium gas through the glass tube.

Then, the above sintering step and collapsing step are repeatedly performed 8 times and 15 times, respectively, at a temperature of 2000°C, thereby obtaining the optical fiber preform doped with Tm^{2+} ion. The optical fiber preform is drawn to fabricate the optical fiber. Herein, even when a sintering step is carried out at a temperature of 1600 to 2200°C, the same result is also obtained.

A light absorption spectrum of the optical fiber fabricated using the doping solution containing the sucrose as a reducing agent is shown in Fig. 2.

Light absorption spectrum of Fig 2 shows that light absorption spectrum shown at 465nm, 680nm, 785nm, 1210nm, and 1600nm is formed by Tm^{3+} ion, and light absorption spectrum distributed in the broad range of 400nm and 900nm is formed by Tm^{2+} ion.

Comparative example 1

Thulium chloride hexahydrate ($TmCl_3 \cdot 6H_2O$) of 0.04M and aluminum chloride hexahydrate ($AlCl_3 \cdot 6H_2O$) of 0.19M are dissolved in ethanol to prepare a doping solution without containing the sucrose as the reducing agent.

A core layer having a porous fine structure is formed at an inner wall of a silica glass tube in the same method as the embodiment 1. The fabricated doping solution is injected into the glass tube and then discharged after 1 hour. Then, the core layer is dried together with the purge of helium, oxygen and chlorine through the tube.

Then, the above sintering step and collapsing step are repeatedly performed 3 times and 7 times, respectively, at a temperature of 2000°C, thereby obtaining the optical fiber preform doped with Tm^{3+} ion. The optical fiber preform is drawn to fabricate the optical fiber.

A light absorption spectrum of the optical fiber fabricated by using the doping solution without containing the sucrose as the reducing agent is shown in Fig. 3. Differently from the result of the embodiment 1 using the reducing agent, Fig 3 shows only light absorption spectrum according to Tm^{3+} ion.

Embodiment 2

Europium chloride ($EuCl_3 \cdot xH_2O$) of 0.097M and sucrose ($C_{12}H_{22}O_{11}$) of 0.518M are dissolved in deionized water to prepare a doping solution containing

rare earth ion (Eu^{3+}) and the sucrose as a reducing agent.

Then, the optical fiber doped with Eu^{2+} ion is fabricated by the same processes as in the embodiment 1.

A light absorption spectrum of the optical fiber fabricated by using the doping solution containing the sucrose as the reducing agent is shown in Fig. 4. In the light absorption spectrum of Fig. 4, the light absorption spectrum distributed in the broad range of 600nm and 1200nm is formed by Eu^{2+} ion. This spectrum is not shown in the case of Eu^{3+} ion.

Comparative example 2

Europium chloride hydrate ($EuCl_3 \cdot xH_2O$) of 0.097M and aluminum chloride hexahyrate ($AlCl_3 \cdot 6H_2O$) of 0.518M are dissolved in ethanol to prepare a doping solution without containing the sucrose as the reducing agent.

Then, the optical fiber doped with Eu^{3+} ion is fabricated by the same processes as in the comparative 1.

A light absorption spectrum of the optical fiber fabricated by using the doping solution without containing the sucrose as the reducing agent is shown in Fig. 5. Differently from the result of the embodiment 2 using the reducing agent, Fig 5 shows only light absorption spectrum according to Eu^{3+} ion.

The above embodiments 1 and 2 illustrate that the optical fibers perform doped with Tm^{2+} ion and Eu^{+2} ion, respectively, by the doping solutions containing the reducing agents are obtained. Depending on the intensity of reduction potential which the reducing agent has, it is confirmed that metal ion or rare earth ion having 3+ valence is changed to 2+ valence or 1+, in some cases to "0" valence. When the metal ion or rare the earth ion is reduced to "0" valence, an optical fiber

preform or an optical device preform doped with metal particle or rare earth element is formed.

As described above, the present invention can fabricate an optical device doped with reduced metal ion and/or rare earth ion having a desire valence by a facile solution doping technique.

According to the present invention, an optical fiber or an optical device doped with metal ion and/or rare earth ion reduced by the facile solution doping technique, with no need to change the conventional MCVD, VAD, OVD processes, etc. may be fabricated.

While the present invention has been described in detail, it should be understood that various changes, substitutions and alterations can be made hereto without departing from the spirit and scope of the invention as defined by the appended claims.

CLAIMS

1. A method of fabricating an optical fiber or an optical device doped with reduced metal ion and/or rare earth ion, comprising steps of:

forming a partially-sintered fine structure in a base material for fabricating the optical fiber or the optical device;

soaking the fine structure into a doping solution containing a reducing agent together with metal ion and rare earth ion during a selected time;

drying the fine structure in which the metal ion and/or rare ion are/is soaked; and

heating the fine structure such that the fine structure is sintered.

2. The method of claim 1, wherein the reducing agent is hydrocarbon compounds.

3. The method of claim 2, wherein the hydrocarbon compound is any one selected from the group consisting of glucose, sucrose, glycerine, dextrin, benzene, phenol, hexane, toluene, stylene, and naphthalene.

4. The method of claim 1, where the reducing agent is alkoxide compounds.

5. The method of claim 4, wherein the alkoxide compound is any one selected from the group consisting of TEOS (tetraethyl orthosilicate), TMOS

(tetramethyl orthosilicate), TEOC (tetraethyl orthocarbonate), and TMOC (tetramethyl orthocarbonate).

6. The method of any preceding claim, wherein the metal ion and/or rare earth ion is at least one selected from the group consisting of Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Al, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Y, Zr, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, In, Sn, Hf, Ta, W, Re, Os, Ir, Pt, Au, Tl, Pb, Bi and a mixture thereof.

7. The method of any preceding claim, wherein a base material for fabricating the optical fiber or the optical device has a basic composition comprising a silicon oxide or a composite oxide of a silicone oxide and an oxide; in which the oxide is at least one selected from the group consisting of germanium oxide (GeO_2), boron oxide (B_2O_3), phosphorous oxide (P_2O_5), and titanium oxide (TiO_2).

8. The method of any one of claims 1 to 6, wherein a base material for fabricating the optical fiber or the optical device has a basic composition selected from silica (SiO_2), germanosilicate (SiO_2-GeO_2), phosphorosilicate (SiO_2-P_2O_5), phosphorogermanosilicate (SiO_2-GeO_2-P_2O_5), borosilicate (SiO_2-B_2O_3), borophosphorosilicate (SiO_2-P_2O_5-B_2O_3), borogermanosilicate (SiO_2-GeO_2-B_2O_3), titanosilicate (SiO_2-TiO_2), phosphorotitanosilicate (SiO_2-TiO_2-P_2O_5), or borotitanosilicate (SiO_2-TiO_2-B_2O_3).

9. The method of any preceding claim, wherein, the step of forming the partially-sintered fine structure in the base material is performed by a process selected from the group consisting of MCVD (modified chemical vapor deposition), VAD (vapor-phase axial deposition), VOD (outside vapor deposition), and FHD (flame hydrolysis deposition).

10. A method of fabricating an optical fiber or an optical device doped with reduced metal particle and/or rare earth element, comprising steps of:

forming a partially-sintered fine structure in a base material for fabricating the optical fiber or the optical device;

soaking the fine structure into a doping solution containing a reducing agent having strong reduction potential together with metal ion and rare earth ion during a selected time,

drying the fine structure in which the metal ion and/or the rare earth ion is/are soaked; and

heating the fine structure such that the fine structure is sintered, thereby forming the metal particle and/or the rare earth elements.

11. The method of claim 10, wherein the reducing agent is hydrocarbon compounds.

12. The method of claim 10, where the reducing agent is alkoxide compounds.

Application No: GB0421656.0 **Examiner:** Kathryn Orme

Claims searched: 1-12 **Date of search:** 24 February 2005

Patents Act 1977: Search Report under Section 17

Documents considered to be relevant:

Category	Relevant to claims	Identity of document and passage or figure of particular relevance
X	1-2, 6-7, 9, 10 and 11 at least	EP 0517955 A1 (CORNING INC) see especially column 3 lines 7-56 and column 4 line 45
X	1-3, 6-7 and 10-11 at least	US 4610708 A (SARHANGI ET AL.) see especially the abstract and column 5 lines 24-31
X	1, 4-7, 9-10 and 12 at least	WPI Abstract Accession No. 1991-243530 & JP 3159924 (Fujikura Ltd) 09/07/1991 (see abstract)
X	1-2, 6-7 and 9-11 at least	WPI Abstract Accession No. 1991-019603 & JP 2293332 (Shinetsu Chemical et al.) 04/12/1990 (see abstract)

Categories:

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

Field of Search:

Search of GB, EP, WO & US patent documents classified in the following areas of the UKC^{X} :

C1M

Worldwide search of patent documents classified in the following areas of the IPC^{07}

C03B; C03C

The following online and other databases have been used in the preparation of this search report

WPI and EPODOC