# PREPARATION OF PREFORMS AND OPTICAL FIBRES CONTAINING ALUMINIUM BY THE SOLUTION--DOPING METHOD

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This paper is concerned with the alternative dopant  $Al_2O_3$  for Modified Chemical Vapour Deposition solution-doping silica based optical fibre. An extension of the solution-doping technique is described by which all processes out of deposition line are removed and organic solution is used. The refractive index profile, spectral attenuation, and cutoff wavelength have been investigated. The fabricated single mode fibres show nearly triangular and step-index profile and spectral attenuation below 10 dB/km in region between 950–1300 nm and at 1550 nm.

#### INTRODUCTION

Optical fibres are light guides with extremely low attenuation. These guides are formed by a central core of slightly higher refractive index than the coaxial outer cladding. Depending on wavelength, refractive index difference, refractive index profile, and core diameter, the fibre can guide one or more modes of the electrical and magnetic field. Single mode fibres are the commonest choise for transmission on trunk and subscriber lines. The waveguide core is usually formed by a few coaxial layers composed of  $GeO_2$ —SiO<sub>2</sub> glass and the optical cladding of F—P<sub>2</sub>O<sub>5</sub>—SiO<sub>2</sub> glassy layers. Present production processes are based on various CVD (chemical vapour deposition) methods, both inner and outer techniques.

Germanium oxide is still the most widely used dopant in silica-based optical fibres to raise the refractive index. Lately many materials have been proposed as alternative dopants, especially aluminium oxide is considered to be very promising and used one [1, 2]. In regard of higher temperature of sublimation of aluminium chloride to boiling point of germanium chloride (raw materials for preform preparation), the VAD (vapour axial deposition) method [3] and the solution-doping technique [4, 5] are used especially. Previous works with the solution-doping technique have concentrated on soaking of a partially-sintered core layers in an aqueous solution of aluminium chloride.

In this paper an extension of the solution-doping technique is described by which all processes out of deposition line (in MCVD set up) have been removed and aqueous solution by organic one has been replaced. The technique may also be used to incorporate many further dopants into optical fibres, for instance rare-earth elements.

## EXPERIMENTAL

Preforms for optical fibres containing aluminium oxide have been prepared by the MCVD (modified chemical vapour deposition) method.

The MCVD process is based on the high temperature oxidation of reactant gases inside a rotating tube which is heated by an external source. Controlled quantities

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of the reactants are transported to the reaction zone by passing dry oxygen through the liquid precursors, usually halides.

In our technique a conventional  $F_2O_5$ —SiO<sub>2</sub> optical cladding from gaseous phase was prepared. The core layers were deposited in two steps: (I) a partially-sintered porous soot of SiO<sub>2</sub> or of SiO<sub>2</sub>+P<sub>2</sub>O<sub>5</sub> was formed at a reduced temperature; (II) the tube with porous layer was contracted in two points (P<sub>1</sub> and P<sub>2</sub>) to form a tub-like reaction chamber (Fig. 1), into which the dopant solution was injected and evaporated while the tube was rotating and oxygen flowing through the tube. The deposit was dried, sintered and the whole tube collapsed in an O<sub>2</sub>/CCl<sub>4</sub> atmosphere.



Fig. 1. Schematic view of low-vapour-pressure dopants technology deposition by the modified solution-doping method: a - silica tube, b - porous layer, c - organic solution of dopant,  $P_{1,2} - reaction$  chamber margins.

Preforms were fabricated using trichloromethane solutions of strengths up to 0.01 molar AlCl<sub>3</sub>. Heralux WG tubes ( $18 \times 1.4$  mm), SiCl<sub>4</sub>, POCl<sub>3</sub> (F.O. Optipur), AlCl<sub>3</sub>, CHCl<sub>3</sub>, SF<sub>6</sub> (P.A. quality) were used. The refractive index profile of preforms and fibres was measured by the commercial equipment P 101 and S 14 (York Technology). Single mode fibres with diameters 125  $\mu$ m were drawn from the preforms and covered with UV curable acrylate. The spectral attenuation and cutoff wavelength were measured using the S 15 device (York Technology). The concentration of aluminium oxide was estimated from the refractive index profile of the preform on base of knowledge of the refractive index dependence on the glass composition.

### **RESULTS AND DISCUSSION**

Preforms with  $F_{--}P_2O_5$ —SiO<sub>2</sub> optical cladding and core composed of  $Al_2O_3$ — SiO<sub>2</sub> or of  $Al_2O_3$ — $P_2O_5$ —SiO<sub>2</sub> glass (aluminium oxide concentration up to 2 mole %) were prepared. By the reason that aluminium oxide solubility in trichloromethane is low, the higher refractive index difference would be problematical. Fig. 2 shows the refractive index profile of the fabricated single mode fibre with the core composed of  $Al_2O_3$ —SiO<sub>2</sub> glass. The index profile is like triangular course and relative refractive index difference between the core and silicon oxide  $\Delta n^+$  is 0.0023. Nearly step index profile illustrated in Fig. 3 was measured in the fibre with  $Al_2O_3$ — $P_2O_5$ —SiO<sub>2</sub> core. The relative refractive index difference  $\Delta n^+$  is 0.0027 here. Fig. 4 shows the spectral attenuation of a fibre characterized by the 125  $\mu$ m outer diameter, 9.8  $\mu$ m core diameter, 0.0027 relative refractive index difference between core and cladding, and a deposited cladding/core diameter ratio 6.6. The cutoff wavelength is 920 nm. The best attenuation value of the fibres drawn from these preforms was in region between 950-1300 nm and at 1550 nm below 10 dB/km. The relative high attenuation can be explained for instance by the high level of OH groups and that matter of fact is in drying process.



Fig. 2. Refractive index profile of single mode fibre with core composed of Al<sub>2</sub>O<sub>3</sub>—SiO<sub>2</sub> glass.



Fig. 3. Refractive index profile of single mode fibre with  $Al_2O_3$ — $P_2O_5$ — $SiO_2$  core.





Fig. 4. Spectral attenuation of single mode fibre with refractive index profile as in Fig. 3.

From the experiments, it may be concluded that our solution-doping technique using the organic solution is determined for the single mode fibres preparation with various index profile course. The fabricated single mode fibres show comparable optical properties with fibres prepared by other workers by the MCVD method [2, 4, 5]. The process developed in our institute is suitable especially for the rare-earths (high concentration) doped, single mode fibres used in devices, sensors, and in fibre amplifiers.

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#### PŘÍPRAVA PREFOREM A OPTICKÝCH VLÁKEN OBSAHUJÍCÍCH HLINÍK METODOU "SOLUTION-DOPING"

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Byla připravena optická vlákna se světlovodným jádrem s oxidem hlinitým modifikovanou metodou depozice z plynné fáze (MCVD) rozšířenou o techniku "solution-doping". Tato technika byla upravena pro použití dopantů rozpuštěných v organickém rozpouštědle a byly odstraněny veškeré postupy prováděné mimo zařízení MCVD. Na jednovidových vláknech byl zkoumán profil indexu lomu, útlum a mezní vlnová délka. Připravená vlákna mají téměř trojúhelníkový a step-indexový profil indexu lomu, útlum v oblasti 950–1300 a 1550 nm pod 10 dB/km.

- Obr. 1. Schéma depozice dopantů s nízkou tenzí par upravenou metodou "solution-doping": a křemenná trubice, b – porézní vrstva, c – roztok dopantů v organickém rozpouštědle, P<sub>1,2</sub> – okraje reakční komory.
- Obr. 2. Profil indexu lomu jednovidového vlákna s jádrem o složení Al<sub>2</sub>O<sub>3</sub>—SiO<sub>2</sub>.
- Obr. 3. Profil indexu lomu jednovidového vlákna s jádrem o složení Al<sub>2</sub>O<sub>3</sub>-P<sub>2</sub>O<sub>5</sub>-SiO<sub>2</sub>.
- Obr. 4. Závislost optických ztrát na vlnové délce jednovidového vlákna s profilem indexu lomu znázorněném na obr. 3.

## ПОЛУЧЕНИЕ ЗАГОТОВОК И ВОЛОКОННЫХ СВЕТОВОДОВ С СОДЕРЖАНИЕМ АЛЮМИНИЯ С ПОМОЩЬЮ МЕТОДА SOLUTION-DOPING

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Были получены волоконные световоды со световодной сердцевиной, содержащей оксид трехвалентного алюминия, с помощью модифицированного метода химического парофазового осаждения (MCVD), дополненного техникой solution-doping. Приводимая техника была приспособлена для использования легирующих компонентов, растворенных в органическом растворителе и были исключены все проводимые способы кроме MCVD. В случае одномодовых световодов исследовали профиль показателя преломления, потери и длину волны отсечки. Полученные волокна имеют почти треугольный и скачкообразный профиль показателя преломления, потери в диапазоне 950–1300 и 1550 nm ниже 10 dB/км.

- Рис. 1. Схема осаждения легирующих компонентов с низкой упругостью насыщенного пара с помощью модифицированного метода solution-doping: а – кварцевая трубка, b – пористый слой, с – раствор легирующих компонентов в органическом растворителе, P<sub>1,2</sub> – краи реакционной камеры.
- Рис. 2. Профиль показателя преломления одномодового световода со сердцевиной составом Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>.
- Рис. 3. Профиль показателя преломления одномодового световода со сердцевиной составом Al<sub>2</sub>O<sub>3</sub>-P<sub>5</sub>-SiO<sub>2</sub>.
- Рис. 4. Зависимость оптических потерь от длины волны одномодового световода с профилем показателя преломления, изображаемого на рис. 3.