Optical Fiber Technology 28 (2016) 23–27

Contents lists available at ScienceDirect

Optical Fiber Technology

www.elsevier.com/locate/yofte

Regular Articles

Solution doped preform with improved uniformity and concentration using dual-layer soot deposition

S.Z. Muhd-Yassin [a,*], Nasr Y.M. Omar [b], K.A. Mat-Sharif [a], M.I. Zulkifli [a], M.H. Safar [a], S.M. Aljamimi [b], Z. Yusoff [b], S.D. Emami [b], M.C. Paul [c], H.A. Abdul-Rashid [b]

[a] Photonics Laboratory Telekom Malaysia Research and Development, Cyberjaya, Selangor, Malaysia
[b] Faculty of Engineering, Multimedia University, Cyberjaya, Selangor, Malaysia
[c] Fiber Optics and Photonics Division, Central Glass and Ceramic Research Institute (CGCRI), Council of Scientific and Industrial Research (CSIR), Jadavpur, Kolkata 700032, India

ARTICLE INFO

Article history:
Received 20 March 2015
Revised 16 July 2015
Available online 21 January 2016

Keywords:
MCVD
Solution doping technique
Porous silica
Optical fibre
Uniformity

ABSTRACT

A new method of soot deposition to improve the characteristics of a solution-doped optical fibre preform is reported. The soot was generated using a modified chemical vapour deposition (MCVD) technique. A better longitudinal uniformity of the core refractive index profile and a higher degree of dopant incorporation were obtained when two layers of soot were deposited in the core. These improvements were further extended by depositing each layer at different temperatures. A variation of 0.15×10^{-2} (%RSD 3.5%) in the refractive index difference along 23 cm of the preform and a core-to-cladding refractive index difference of approximately 0.012 were achieved using a 1.2 M $AlCl_3$ solution.

1. Introduction

The incorporation of rare earth (RE) elements and glass modifiers into silica fibres has been widely developed to meet the demands of laser and amplifier applications [1]. The introduction of glass network modifiers, such as aluminium or sodium, is known to break the silica network and result in a loose structure, which facilitates the incorporation of RE ions into the network [2]. In several cases, the modifier ions can also enhance the quantum efficiency of RE ion emissions and alter its absorption or emission spectra [2,3]. The precursors of these materials have low vapour pressures that necessitate the use of a separate platform if they are to be used in a standard MCVD process [4]. To date, the incorporation of these materials into silica is mainly achieved by vapour or solution based doping methods [5,6]. The vapour phase techniques offer promising advantages over the solution-doping technique, such as the capability to fabricate complex design fibres, high longitudinal uniformity, and high dopant concentration [6]. However, the fact that these advantages of the vapour phase techniques have yet to be fully realised currently makes the solution-doping technique the preferred doping method.

The solution-doping technique has been widely implemented since the development of erbium-doped fibre (EDF) by Payne et al. [7]. This technique, however, suffers from uniformity and reproducibility issues due to the inconsistent morphology and fragile nature of the solution-impregnated soot layer. The dopant uniformity has been observed to deteriorate at high dopant concentrations [8,9]. In addition, at the same solution strength, Kirchhof et al. [10] reported that the concentration of dopants retained in the soot layer decreases exponentially as the soot density increases. In addition, the soot density has been observed to change linearly with the soot process temperature (i.e., soot deposition temperature) [11]. The dopant concentration is therefore more sensitive to temperature variations at lower soot deposition temperatures. This behaviour restricts the dopant concentration to a lower limit if a considerable uniformity is to be achieved. To increase the uniformity, Atkins and Windeler [9] suggested the use of an internal heat source gas, which results in the formation of an additional soot layer that decreases the sensitivity of the soot-to-temperature variations. However, in their study, the overall uniformity of the final preform was not demonstrated, and other factors, such as external forces, that may affect the uniformity of the final preform were not included. In addition, Kirchhof et al. [10] reported the use of higher density soot, which is deposited at a higher temperature, to enhance the longitudinal uniformity. However, increasing the soot density decreases the dopant concentration due to the reduction of the soot porosity.

In addition to the process temperature, the uniformity of the final preform is dependent on the disruptive external forces that

* Corresponding author.
E-mail address: shahrinzen@tmrnd.com.my (S.Z. Muhd-Yassin).

http://dx.doi.org/10.1016/j.yofte.2016.01.003
1068-5200/

Table 1
Observations during the formation of the soot and the surface area and pore volume of the soot deposited at various temperatures.

Temperature (°C)	Observation	BET surface area (m^2g^{-1})	Pore volume (cm^3g^{-1})
1550	No deposition	NA	NA
1600	White cloud	NA	NA
1650	Soot deposited	31.5	0.1454
1700	Soot deposited	24.6	0.1014
1750	Soot deposited	18.7	0.0558
1800	Soot deposited	11.3	0.0288
1900	Partially sintered	NA	NA
2100	Fully sintered	NA	NA

the deposited soot layer encounters during the multistep solution-doping process. These external forces may be introduced during tube cutting, removal from the lathe, soaking, draining, drying, and remounting on the lathe. Additionally, a higher degree of force may be exerted on the soot if it is impregnated with viscous solutions [14].

In this paper, we report on the improvement of the longitudinal uniformity by depositing multiple soot layers at an appropriate temperature. A 0.15×10^{-2} variation (%RSD 3.5%) in the longitudinal refractive index difference with a relatively high dopant concentration was achieved. In addition, a number of doped preforms, which were fabricated using the conventional single-layer soot at different deposition temperatures, are presented for comparison.

2. Experimental setup

2.1. Determination of the soot deposition temperature range

All of the MCVD experiments were conducted using a Heraeus F300 quartz substrate tube (25 mm OD and 19 mm ID). After rinsing the tube with isopropanol/acetone to remove organic contaminations, it was mounted onto the lathe and heated using an oxy-hydrogen traversing burner. The surface impurities on the tube inner wall were etched using SF_6 gas at 2050 °C with a flow of oxygen. Several layers of pure silica were then deposited and sintered to avoid contaminating the soot with fluorine. For soot deposition, $SiCl_4$ vapour from a bubbler system heated at 34 °C was carried to the substrate tube using 100 sccm of O_2. In addition, 700 sccm of O_2 and 75 sccm of He were flowed simultaneously to promote $SiCl_4$ oxidation and improve the heat distribution in the hot zone, respectively. Concurrently, the substrate tube surface temperature was initially held at 1550 °C by an oxy-hydrogen burner moving at the speed of 125 mm/min in the same direction as the gas flow (i.e., forward deposition). At this deposition temperature, the occurrence of a soot layer was observed, and the deposited soot was subsequently sintered in the next pass. The above soot deposition step was repeated several times at a 50 °C increment in temperature each time until the soot started to exhibit some degree of transparency.

2.2. Fabrication of the preforms using single- and dual-layer soot at different soot deposition temperatures

Five preform tubes were prepared using the same above-mentioned procedures, but with different soot deposition temperatures and different numbers of deposited layers. One layer of soot was deposited inside three different tubes at 1650 °C, 1750 °C and 1800 °C, which were labelled as preform tubes 1, 2 and 3, respectively. In addition, two layers of soot were deposited inside preform tube 4 at 1800 °C by traversing the burner across the tube twice in the forward direction. These steps were repeated for preform tube 5 by setting the soot deposition temperature to 1700 °C and 1750 °C for the first and second deposition passes, respectively. Some of the resultant soot was collected from preform tubes 4 and 5 and then subjected to SEM morphology analysis. For all of the preform tubes, the deposited soot was soaked for 90 min with a 1.2 M $AlCl_3{\cdot}6H_2O$/0.02 M $TmCl_3{\cdot}6H_2O$ ethanol:water (9:1) solution. After the solution was drained from the tube, the soot was dried by flowing a gentle stream of nitrogen gas for one hour. The tube was then heat treated in a tube furnace, where the temperature was raised to 800 °C at a rate of 5 °C/min. This temperature was maintained for 30 min, and then, the tube was allowed to cool down to room temperature naturally. Next, the tube was remounted onto the lathe and heated at 1500 °C while flowing O_2 at 1000 sccm to the tube to oxidise any precursor salts that might remain in the preform tube. The doped silica soot was then slowly sintered with six burner passes starting from 1600 °C and up to 2100 °C in 100 °C increments. Subsequent to sintering, the tube was collapsed to a solid preform in a conventional manner [6].

2.3. Characterisation of the soot and the preforms

For all of the fabricated preforms, some of the deposited soot was scraped off of the substrate tube wall and then analysed for its surface area and pore volume using a surface area and porosimetry analyser (ASAP 2020, Micromeritics). In addition, the soot that was still attached to the substrate tube wall was examined using a scanning electron microscope (SEM) (S-3400N, Hitachi). The refractive index profiles (RIPs) of all of the fabricated preforms were measured using a refractive index profiler (PK104, Photon Kinetics). The measurements were performed transversely along a 23-cm segment of each preform at a 1.0-cm interval. The reported value of the refractive index difference (Δn) at each longitudinal position is the average of three Δn values measured at three different angles. The amount of aluminium incorporated into preform 5 was determined via energy-dispersive X-ray spectroscopy (EDS) (S-3400N, Hitachi) using point ID analysis (40 points across the preform's core). EDS analyses were performed on polished carbon-coated preform slices having a thickness of ~5 mm. The carbon coating was used to provide a conductive layer to minimise charging effects from the glass during EDS analysis. EDS analyses were performed at approximately the same axial positions as the RIP scans to investigate the relationship between the aluminium concentration and the refractive index difference.

3. Results and discussion

Table 1 presents the observations made during the $SiCl_4$ oxidation at temperatures ranging from 1550 °C to 2100 °C. The soot starts to deposit on the tube inner wall at 1650 °C and becomes fully sintered at 2100 °C. The surface area and pore volume of the soot deposited at 1650–1800 °C are determined by the Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) methods, respectively. The results are also listed in Table 1. The BJH analysis in Table 1 demonstrates that the pore volume of the soot is smaller at higher deposition temperatures. This result is expected for the forward deposition, where the soot particles are likely to experience viscous sintering and the collapsing of the pore structure as the burner heats up the deposited soot to the deposition temperature soon after deposition. Meanwhile, the BET analysis indicated that the surface area of the soot decreases as the deposition temperature increases. This result is due to the increase in particle size as more particles fuse together at higher temperatures. The particle size increment with temperature is in agreement with previous reports [13,14] and is confirmed by the SEM