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MCVD in-situ solution doping process for the fabrication of complex design large core rare-earth doped fibers

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ABSTRACT

We report on the fabrication and characterization of rare-earth doped silica fibers manufactured using an in-situ solution doping technique, which is compatible with conventional modified chemical vapor deposition equipment. The in-situ technique permits significantly more doped layers to be deposited, compared to conventional solution doping, and is directly applicable for the fabrication of large core rare-earth doped fibers suitable for high-power fiber sources. Ytterbium-doped aluminosilicate fibers fabricated using the new in-situ doping technique are reported and the results are compared to fibers fabricated using a conventional solution doping method.

1. Introduction

Fibers fabricated for laser or amplifier applications require the core of the preform to be doped with active ions. For many high-power applications ytterbium (Yb) doped fibers are the preferred choice. Yb^{3+} ions have broad emission (typically 975–1200 nm), a long lifetime in the excited state, and can be incorporated in the silica host in relatively high concentrations [1].

Achieving high output powers in fibers has been made possible due to advances in fiber design and fabrication. Fiber structures such as large mode area (LMA) fibers, large flattened mode (LFM) fibers, and fibers with a pedestal geometry [2–5], have all been instrumental in further power-scaling of fiber lasers. Optimizing the wave-guiding structure in these ways typically requires the fabrication of fibers with a large core diameter and increasingly complex doping profiles. For both these prerequisites, the preform fabrication process needs to be capable of depositing ten's of actively doped layers in a controllable manner.

For fiber preforms fabricated using modified chemical vapor deposition (MCVD) [6], it is not possible to deposit rare earth (RE) ions, such as Er^{3+} and Yb^{3+}, using a conventional bubbler system owing to the low vapor pressure of RE halide precursors at room temperature. Similar problems are encountered when co-doping with Al which is required to increase the solubility of the RE ions in the host glass [7]. To overcome these issues an extension of the MCVD process known as solution doping [8–10], is used. Here, at the core deposition stage, a porous oxide layer is deposited on the inside of the silica substrate tube using a reduced burner temperature. The substrate is then separated and the glassware removed from the preform-making lathe and soaked vertically in a solution containing the RE salts and co-dopants dissolved in a solvent. After approximately 1 h the solution is drained and the residual solvent left to evaporate. The tube is then reassembled on the lathe, the doped oxide layer is sintered, and the substrate is collapsed into a preform.

Since its conception the solution doping technique has been the adopted method for incorporating RE and alumina dopants into the preform owing to its simplicity and low cost to implement. The major drawback, however, comes about when multiple layers are required in the preform. The repeated removing and reassembling of the substrate reduces the length of usable preform as the glassware must be re-welded when it is returned to the lathe. There is also the potential for introducing contamination and for disturbing the fragile soot when transferring the glassware between apparatus. When combined with the duration needed for the solution to fully soak the soot layer, the process becomes very time consuming and only appropriate for the deposition of 2–4 layers.

In this paper we report on a modified solution doping technique which eliminates the need to remove, and reassemble, the

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glassware from the lathe thus reducing the process time for depositing each doped layer by 1–2 h compared to conventional solution doping. The in-situ method is ideal for large core fibers and tens of layers can be easily deposited in a reasonable timescale.

2. Experimental procedure

2.1. In-situ solution doping process

The fiber preforms are fabricated on conventional MCVD equipment. A high-quality F300 (Heraeus) silica tube measuring a nominal 20 × 16 mm in diameter and 500 mm in length is used as the starting substrate. The glass is gas-phase etched and then undoped silica cladding layers are deposited in the usual manner. For the core layer, an undoped oxide layer is deposited at a sufficiently low temperature to ensure good porosity. A temperature of between 1550 and 1650 °C is typical depending on precursor flow and the desired layer thickness. Instead of removing the substrate tube from the lathe, as in conventional solution doping, the dopants are introduced into the porous soot with the glassware in-situ as shown in Fig. 1.

A small diameter glass tube is positioned into the tailstock end of the glassware and the other end of the delivery tube is attached to a peristaltic pump via a plastic hose. A bespoke adapter is used on the tip of the glass tube to avoid disturbing the soot layer. The dopant chlorides are dissolved in high purity methanol and are slowly fed through the delivery tube to the soot layer at a rate of 7–15 ml min^{-1} depending on the thickness of the soot layer. Once the layer is completely soaked along the length of the preform, the delivery tube is retracted, and the solvent left to evaporate leaving the dopant ions behind. During the filling process the glassware is not filled completely as in the case of vertical solution doping, but instead the oxide soot is soaked with a thin layer of the dopant solution which is not left to soak but evaporates readily. The tube is kept rotating during and after filling with the addition of an inert gas flow of ~2 L min^{-1} whilst the oxide layer is drying. Depending on the layer thickness and the solution concentration the drying time is approximately 30 min. Once completely dried the doped oxide layer is sintered and the tube is collapsed and sealed in the usual manner.

2.2. Preform fabrication

Several preforms have been fabricated using the in-situ technique in order to refine the process and to optimize the conditions for introducing the solvent, soaking, and drying of the soot. This has enabled the existing recipes, designed for vertical solution doped preforms, to be transferred.

Multiple layers can be deposited in a reasonable timescale using the in-situ technique which has been demonstrated with an Yb/Al doped LMA structure. The preform consists of 10 individual layers which were deposited using a $SiCl_4$ flow of 100 ml min^{-1}, at a burner temperature of 1475 ± 3 °C, and doped with a solution containing Yb/Al. The manufacturing process has been optimized to eliminate any 'central dip'. The preform refractive index profile (RIP) was acquired using a PK2600HP Preform Analyzer (Photon Kinetics) which has a refractive index measurement accuracy of ±0.0002 and is compared against a traceable silica calibration rod (see Fig. 2). The average core NA of the preform is 0.07 and the core diameter is 2.6 mm as measured at the full width half maximum (FWHM). The preform RIP shows a refractive index variation in the core and is presented in a way so that the individual layers can be distinguished. Fiber drawn from this preform, however, confirms that this variation is reduced to less than 1×10^{-4} as measured by an S14 Refractive Index Profiler (Photon Kinetics). The process time to deposit 10 Yb/Al doped layers was around 8 h, in addition to the standard time taken to deposit the initial cladding layers and collapse the preform.

To directly compare the in-situ and conventional solution doping techniques, identical preforms with one solution doping step were fabricated using each process. A high-tolerance F300 substrate tube measuring 20 × 16 mm in diameter and 500 mm long was used for each preform. A silica soot layer was deposited using a burner temperature of 1550 ± 3 °C for both preforms to ensure identical soot porosity and the same solution strength of $AlCl_3{\cdot}H_2O$ and $YbCl_3{\cdot}H_2O$ dissolved in methanol was used in each case. Preform A was fabricated using the in-situ technique. Preform B was fabricated using the conventional solution doping method and consisted of 1 h vertical soaking and 1 h vertical drying. The solution for Preform A was fed into the substrate tube at a feed rate of 10–12 ml min^{-1} until the soot was soaked. After solution infiltration the oxide layer was subject to 30 min drying with a flow of nitrogen. The tube remained on the lathe throughout the process. The RIP of the collapsed preforms can be seen in Fig. 3. Preform A has a core NA of approximately 0.17 and Preform B has a core NA of 0.13, as measured from the graph. The core diameter of Preform A and B is 0.99 and 0.86 mm, respectively, measured at the FWHM. The variation in refractive index along the length is less than 5×10^{-4} for each preform. The slight difference in the core diameter is attributed to an increase in the

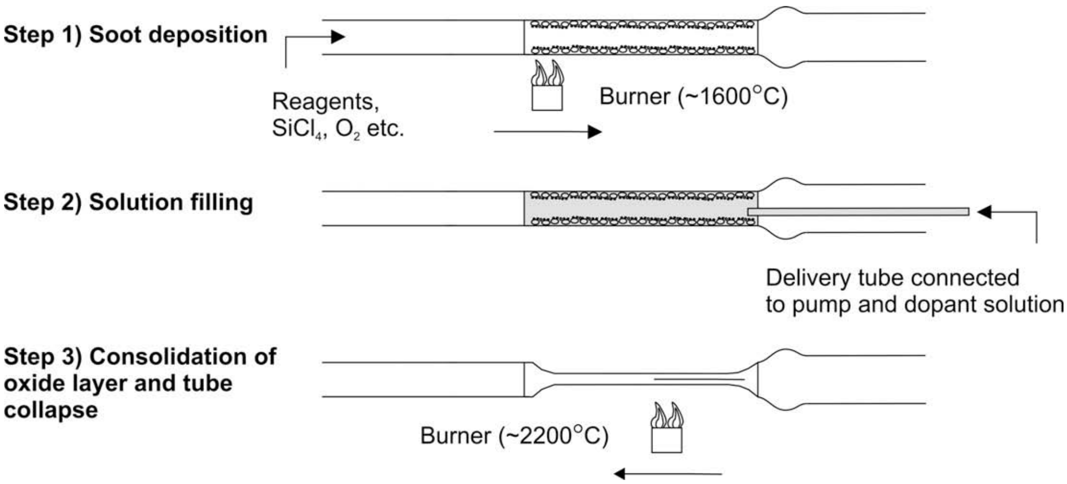

Fig. 1. Process steps for in-situ solution doping.