Optical Fiber Fabrication Using Novel Gas Phase Deposition Technique

Alexander J. Boyland, Andrew S. Webb, Seongwoo Yoo, Francesca H. Mountfort, Mridu P. Kalita, Robert J. Standish, Jayanta K. Sahu , David J. Richardson and David N. Payne

***Abstract*— We report a highly versatile chemical-in-crucible preform fabrication technique suitable for gas-phase deposition of doped optical fibers. Aluminosilicate and ytterbium-doped phosphosilicate fibers are presented demonstrating the technique and its potential for realizing complex fiber designs which are suitable for the next-generation of high power fiber devices. The results show aluminum doped fiber with NA of 0.28 and ytterbium-doped fiber with a measured slope efficiency of 84 % with respect to pump launch power.**

***Index Terms*—Fabrication, Fiber lasers, MCVD, Optical fiber fabrication, Rare earth metals**

I. Introduction

Optical fibers fabricated for laser or amplifier applications require the core to be doped with active ions. For many high-power applications ytterbium (Yb) doped fibers are the preferred choice as Yb^{3+} ions have a broad emission (typically 975 – 1200 nm), a long lifetime in the excited state, and can be incorporated in to the silica host in relatively high concentrations [1]. The ability to incorporate rare-earth (RE) ions into the glass preform in the gas-phase offers the potential for realizing complex preform designs.

Unlike the conventional reagents used in modified chemical vapor deposition (MCVD) the equivalent RE halide compounds are significantly less volatile at room temperature and require several hundred degrees to evolve sufficient vapor [2]. Over the years numerous gas-phase deposition methods using RE precursors have been proposed, such as the 'heated frit' and 'heated source' [3-4], which aim to position the dopant precursor close to the reaction zone so as to avoid the vapor condensing during transportation. Difficulties with accurately heating of the dopant to high temperatures (> 800 ºC) and reactions with the reagents occurring prior to the deposition zone have meant that these techniques have proved unreliable. The technique of choice has remained the MCVD solution doping technique [5] which has been used in the area of high-power fiber lasers with tremendous success. However, the conventional solution doping approach has now been pushed to the limits and new fabrication techniques are required to allow much higher RE doping concentrations with increased uniformity along the length of the preform, increased core size, high precision in both dopant and refractive index profile, and low background loss in fiber with good optical to optical conversion efficiency – all factors which are critical in taking the high power fiber laser technology forward.

Lanthanide-based chelate complexes offer exciting opportunities to dope preforms with RE ions, in the gas-phase, during core-layer deposition [6]. Their relatively high volatility means that sufficient vapor to achieve high dopant concentrations is possible at a moderately low temperature of around 150 – 200 ºC [7]. In addition, since the incorporation of the RE and modifier ions, such as Al_2O_3 and P_2O_5, takes place simultaneously with the glass formation, there is the possibility of less RE clustering and reduced photodarkening, for a given level of doping, as compared with traditional solution doping. Although gas-phase deposition using chelate complexes has been around for some time and MCVD-based vapor delivery system are commercially available, the reported results in this area have been sparse. This is thought to be a result of the practical difficulties in implementing an external delivery system which requires a complicated arrangement of

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delivery lines heated to around 200 ºC to avoid condensation of the precursors [8]. These systems reportedly suffer from 'blocked' lines and poor preform repeatability.

In this paper we report on a novel, versatile, MCVD-based preform manufacturing technique utilizing chelate compounds, and other moderately low vapor pressure precursors. Instead of an external chemical delivery source the precursor is held in a heated crucible within the MCVD glassware and in close proximity to the deposition zone, thereby avoiding the previously cited problems of transporting the vapor through heated lines. The technique has been termed as chemical-in-crucible (CIC), and enables the temperature of the source material to be accurately controlled ensuring a steady flow of dopant vapor to the substrate tube. It permits multi-layered RE-doped preforms to be fabricated as well as providing a means for a range of low-volatility precursors to be prototyped for their suitability in preform manufacture. The approach will allow for the rapid production of preforms with a large volume of doped core.

Fabrication and characterization of a high-NA (0.28) Al-silicate fiber, and a highly ytterbium doped phosphosilicate fiber, both obtained using the CIC technique are presented here.

II. Chemical-in-crucible technique

A schematic of the chemical-in-crucible technique used for preform fabrication is shown in Fig 1. The RE, or other precursors with low volatility at room temperature are placed in a glass crucible which is positioned in close proximity to the substrate tube.

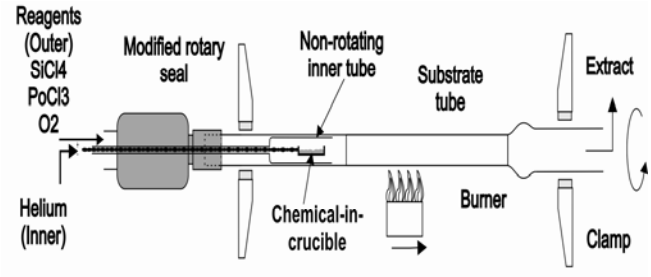

Fig. 1. Schematic of chemical-in-crucible technique for MCVD preform fabrication

The crucible is electrically heated by an external power supply and a thermocouple monitors the temperature so that it can be maintained to within +/- 1 ºC of the set point through a feedback control loop. For use with chelate compounds the crucible is heated in the range of 150 – 300 ºC. The specific crucible shape and size is critical to the dopant 'pick-up' and has been designed such that the vapor flows into the substrate in a steady and uniform manner. The crucible dimensions and capacity also influence the number of layers that can be deposited and their composition. Matching the quantity of dopant to the number of deposited layers required reduces any wastage of chemical, offering a potential cost saving, but more importantly unlike external delivery systems this 'single-use' approach avoids any possibility of thermal degradation of precursors through repeated heating and cooling cycles as the chelate compound may undergo changes of its properties at higher temperatures [9]. This will lead to better preform processing and enhanced reproducibility.

During preform processing the crucible is heated to a temperature at which the chosen chemical dopant will evolve sufficient vapor for the required dopant concentration. This is transported downstream to the reaction zone by the helium carrier gas flowing over the dopant material. These vapors are initially separated from all other bubbler reagent chemicals ($SiCl_4$, $POCl_3$, etc.) used in MCVD system which pass through the outer glassware. Modifications to the standard rotary seal permit the inner tube to remain gas tight and stationary whilst the outer tube is free to rotate. Our approach doesn't require heating of the rotary seal as would be necessary for the alternative external vapor source delivery system.

III. Preform fabrication

Here we report on passive aluminosilicate, and active Yb-doped phosphosilicate fibers fabricated from gas-phase doped preforms using the CIC technique. In both cases a standard step-index profile was targeted. Aluminium chloride ($AlCl_3$), of 99.99% purity, was used as a precursor for aluminium oxide (Al_2O_3), which sublimes at temperatures above 100 ºC. The starting material for fabricating the Yb-doped fiber was organometallic chelate tris(2,2,6,6-tetramethyl-3,5-heptanedionato)ytterbium, abbreviated to $Yb(tmhd)_3$, with a purity of 99%. It has a melting point at around 168 ºC and a vapor pressure of around 1kPa at a temperature of 200ºC [6]. Preforms were fabricated using Heraeus F300-quality substrate measuring a nominal 20 mm outer x 16 mm inner diameters with the glassware arranged as depicted in Fig. 1. The conventional glass preparation steps and cladding layers were applied in each case prior to deposition.

The Al-doped preforms were fabricated using the CIC technique. $AlCl_3$ placed in the crucible was heated to a temperatures ranging of between 130-200ºC during the core deposition passes. Helium carrier gas flow rates were varied between 500-2000 ml/min to enable efficient transport of the vapor into the substrate. The burner temperature was ~1950ºC. Following deposition the substrate tube was collapsed and sealed. The Yb-doped phosphosilicate preform was fabricated in the same way as described for the Al-silicate preform. The core layers were deposited using a burner temperature of ~1800ºC and crucible, containing the $Yb(tmhd)_3$, was heated between 200-250ºC for varying Yb concentration. The helium gas flow was directed over the crucible whilst a ratio of 3:1 was used for the $POCl_3$:$SiCl_4$ flows. Following deposition the tube was collapsed and sealed.

IV. Characterization and Results

A. Aluminosilicate fiber

The refractive index profile (RIP) of the preforms was measured using a PK2600HP Preform Analyzer (Photon Kinetics). The RIP trace obtained for an Al-silicate preform can be seen in Fig. 2. The preform RIP was measured at every 25 mm along the length of the preform and showed good uniformity with less than 10% variation over 200mm. The NA is 0.28 with a crucible temperature of 175ºC, and goes up to 0.31 when the temperature is increased to 190ºC. As aluminum is the only dopant contributing to the refractive index increase, the profile uniformity along the preform length gives an accurate representation of the dopant uniformity.

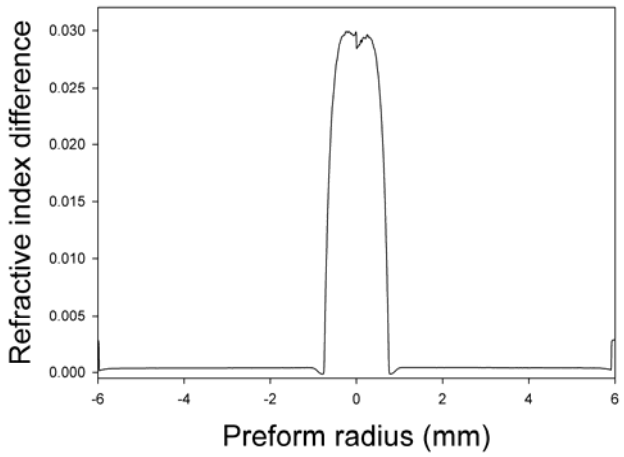

Fig. 2. Refractive index profile of Al-silicate preform fabricated using CIC technique

The preform was drawn with a core size of 10µm and an outer diameter of 125µm and coated with a polymer material of higher index than the silica glass. High resolution OTDR (Lucial v-OTDR) measurement was performed to assess the background loss in the fiber. The attenuation at 1285 nm is 16dB/km, which is the lowest reported value for such high NA in this type of fiber to the best of our knowledge.

B. Yb doped phosphosilicate fiber

A small central dip was observed in the profile, fig.3, as a result of P_2O_5 evaporation during the seal pass as phosphorous over-doping was not performed.

Yb-doped fiber was drawn to 125µm diameter with a 10µm core size in a double-clad (DCF) configuration and characterized using a white-light to assess the background loss and Yb concentration uniformity of core layer. The induced absorption from Yb^{3+} ions was equated to 20600 ppm-wt. OTDR measurements for the background loss were made for all fibers. These ranged from 30-70dB/km at 1285nm dependent on Yb concentration. This higher than expected background loss is attributed to the low purity of chelate precursor used in preform fabrication.

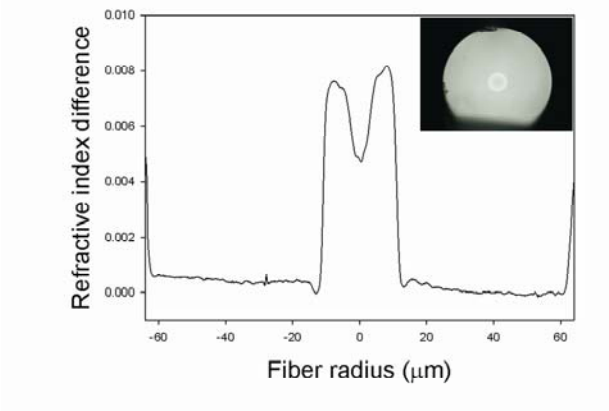

Fig. 3. Refractive index profile of Yb-doped P-silicate fiber fabricated using CIC technique, with photo of fiber inset

Laser efficiency was tested in cladding-pump configuration using a fiber coupled 975 nm laser diode via collimating and focusing lenses. Both ends of the fiber were cleaved perpendicularly to the fiber axis. The 4% Fresnel reflections from both fiber facets formed a linear laser cavity. The output signal was separated from the pump beam using a dichroic mirror at each end. The schematic of this experimental setup is shown in Fig.4. The laser output power characteristics are shown in Fig. 5, together with the output spectrum taken at maximum output power with 1nm resolution. The fiber shows good slope efficiency of 84% with respect to launch pump power. DCF was also drawn to a larger diameter of 400µm with 40µm core size and D-shaped inner cladding to test in high power laser configuration. The output power of the fiber reached 200 W for a launched pump power of just over 250 W [10].

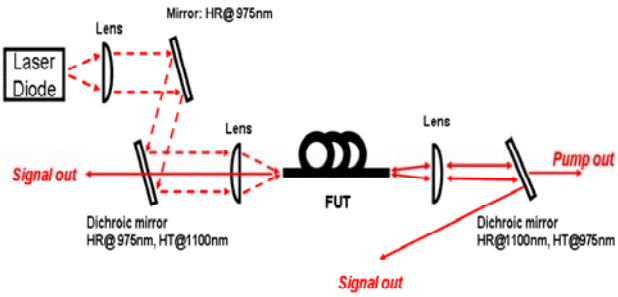

Fig. 4. Schematic of experimental setup for laser efficiency measurement. (FUT: Fiber under test, HR: High reflection, HT: High transmission)

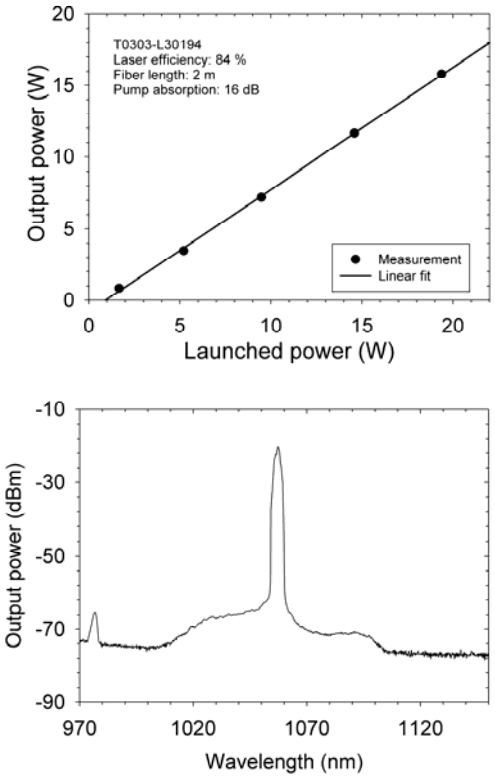

Fig. 5. a) Laser output power *vs* launched pump power in 4%-4% linear cavity configuration and b) Output spectrum at the maximum output power

V. Conclusions

We report a simplified and robust gas phase technique for manufacturing RE doped optical fiber. Results from Yb doped fiber fabricated using this technique exhibited high efficiency showing the potential for this fabrication method. Aluminosilicate fibers with high NA of 0.28 and low background loss are also presented. We have also investigated doping with other dopants using the CIC technique at temperatures exceeding 500ºC which is impractical using external source delivery systems.

References

1. J. Kirchhof, S. Unger, A. Schwuchow, and S. Grimm, "Materials for high-power fiber lasers," J. Non-Cryst. Solids 352, 2399-2403 (2006).
2. S. Sudo, Optical Fiber Amplifiers (Artech House,1997)
3. S. B. Poole, D. N. Payne, M. E. Fermann, and R. I. Laming, "Fabrication of low-loss optical fibers containing rare earth ions," Electron. Lett. 21, 737-738 (1985).
4. M. J. F. Digonnet, "Rare Earth Doped Fiber Lasers and Amplifiers," (Marcel Dekker,1993)
5. J. E. Townsend, S. B. Poole, and D. N. Payne, "Solution-doping technique for fabrication of rare-earth-doped optical fibres," Elect. Lett. 23, 329-331 (1987).
6. R.P.Tumminelli, B.C. McCollum, and E. Snitzer, "Fabrication of high-concentration rare-earth doped optical fibers using chelates," Journal of Lightwave Technology, 8, 1680-1683 (1990)
7. J. E.Sicre, J. T.Dubois, K. J.Eisentraut, R. E.Sievers, "Volatile lanthanide chelates. II. Vapor pressures, heat of vaporisation, and heats of sublimation'', J. Am. Chem. Soc. 91, 3476 (1969)
8. R. J. Mansfield, B. C. McCollum, R.P. Tumminelli "Method for fabricating optical fibers having cores with high rare earth content," US Patent 4 826 288, May 2, 1989.
9. L. Xiong, E. H. Sekiya and K. Saito "Mass fabrication of homogeneously Yb-doped silica nanoparticles and their spectroscopic properties" Nanotechnology, 20, 15605 (2009)
10. A.J.Boyland, A.S.Webb, M.P.Kalita, S.Yoo, C.A.Codemard, R.J.Standish, J.Nilsson, and J.K.Sahu "Rare earth doped optical fiber fabrication using novel gas phase deposition technique" Proceedings of Conference on Lasers and Electro-Optics 2010 paper CThV7

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