

# Benchtop Mini Preform Fabrication for Specialty Optical Fibers

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**Abstract:** We propose and demonstrate a benchtop version of the mini preform and hence short length fiber fabrication system. The system is compact, low cost, fast and flexible compared to the standard fabrication systems. We mimic the recipe used in standard Modified Chemical Vapour Deposition (MCVD) preform fabrication process. Incorporating with solution doping technique, we have fabricated several short length rare earth doped silica fibers, including Ytterbium and Erbium. The results obtained serve as a good indication on the composition of the rare earth elements to be used in the standard processes. The technique is promising and suitable for rapid specialty optical fiber prototyping.

## 1 INTRODUCTION

There are several methods currently available for the fabrication of optical fiber preforms, which can be broadly classified as either vapor or non-vapor based. The vapor based methods include vapor axial deposition (VAD) (Izawa, 2000), outside vapor deposition (OVD) (Petit et al., 2010), (Blankenship and Deneka, 1982), (Cho et al., 1998) and modified chemical vapor deposition (MCVD) (Nagel et al., 1982). The non-vapor based methods are sol-gel processing (Matejec et al., 1997), powder sintering (Auguste et al., 2014), and direct nano-particle deposition (DND) (Tammela et al., 2002). Among these, MCVD process is well established and regularly being employed even for commercial optical fiber production. The process involves passing a mixture of gases through a rotating glass tube which is continuously heated at the surface via a moving burner. Chemical reactions in the gas results in a layer of fine soot being formed on the inner surface of the tube. This subsequently sintered into a clear glass layer, and finally collapsed into the preform to be used for fiber fabrication. High precision of this process enables the production of high quality preforms and fibers. However, it often requires many iterations in order to arrive at the intended fiber design, which increases the cost and time needed for a successful fabrication. It is not surprising that sometimes 30 – 50 iterations may be required for a complete fruitful fabrication. Simply put, preforms

obtained from the earliest iterations are often unsuitable for applications and therefore wasted.

Realistically, the early stages of fiber research involve experimenting with different glass hosts, dopants, mixture of dopants, doping concentrations, geometries, etc. and only require small volumes of fiber. In this work, we propose and demonstrate a scaled-down version of the standard fabrication process for making the mini preforms that can be drawn into short length optical fibers. The short length optical fibers produced are suitable for rapid prototyping, testing and the first order process optimization.

The developed setup, which is a large diameter fiber splicer convertible; is compact and able to reduce the time and cost investment compared to the existing standard fiber fabrication processes. To elaborate, the conventional process yields a 12 mm thick, 300 – 400 mm long silica preform that is used to draw into the required fiber. A single iteration from preform fabrication to fiber pulling takes approximately 2 working days. In contrast, our approach produces ~ 1 mm thick, ~ 20 mm long preforms, which can be drawn into sub-meter length of fibers sufficient for initial testing and analysis. A single iteration of this process takes about 2 – 3 hours. More significantly, the proposed system and process facilitates the analysis and optimization of initial iterations for standard preform fabrications. Conventional preform fabrications can therefore be shortened to reduce excess time, cost and materials

for greater research efficacy. We report in this paper our initial success in fabricating centimeter-long 400  $\mu\text{m}$  size Ytterbium and Erbium doped silica fibers by using the proposed technique. A short comparison between the existing fiber fabrication process and our proposed technique is listed in Table 1.

Table 1: A comparison between the fabrication techniques.

Parameters	Existing Technique	Proposed Technique
Heat source	Oxy-hydrogen burner	Multi-electrode plasma
Isothermic heat zone	Rotating tube	Ring of fire
Process time	Long	Short
Preform diameter	$\geq 12$ mm	$\leq 2.0$ mm, 1 mm for this study
Fiber length	Long	Short
Precision	High	Low to moderate
Background loss	Low	Moderate to high
Cost per iteration	High	Low
Physical footprint	Lab-sized and multi-storey high	Benchtop and modular units

## 2 FABRICATION SYSTEM

We converted our existing three-electrode large diameter splicer (3SAE-LDS) into the main workhorse for this work. In principle, the proposed method is not limited to multi-electrode plasma system, it can also be applied to graphite-based or  $\text{CO}_2$  laser based large diameter fiber splicers.

### 2.1 System Description

The system consists of a three-electrode plasma discharge that provides a narrow isothermic heat zone around the circumference of the intended mini preform. This allows for directed heating of the preform. Instead of rotating the glass tube to achieve uniform heating as in the standard process, we create a ‘Ring of Fire’ around it. Tungsten electrodes, with melting temperature of  $3422^\circ\text{C}$  are selected to give a maximum processing temperature of about  $3000^\circ\text{C}$ , which is sufficient and comparable to the oxy-hydrogen burner used in the MCVD lathe. Three electrodes are orientated in a ‘Y’ configuration so

that the tips form an equilateral triangle. The electrode spacing can vary depending on the size of the plasma required for a given tube dimension. Each electrode is independently modulated 120 degrees out of phase relative to the other electrodes with a high voltage high frequency ( $\sim 30$  kHz) source. An example of a tube within the three-electrode plasma discharge is shown in Figure 1.



Figure 1: Three-electrode plasma discharge for preform fabrication.

### 2.2 Process Description

100 mm long synthetic quartz capillaries or tubes with inner and outer diameters of 0.5 and 1 mm respectively were used throughout this work. These tubes were cleaned in an ultrasonic acetone bath and heat-dried before the start of vapor deposition.

In most MCVD processes, the bubbler technique is used for vapor deposition. A carrier gas, (typically oxygen) is fed through liquid precursors such as  $\text{SiCl}_4$ ,  $\text{GeCl}_4$ ,  $\text{BBr}_3$  and  $\text{POCl}_3$  to generate the respective vapors to be further delivered into the deposition chamber (Oh and Paek, 2012). In our case, we adopted the evaporator method, where no carrier gas is intentionally introduced but instead the pressure inside the precursor container is maintained at positive level, which is controlled by the temperature of the container. Due to the formation of a negative pressure gradient extending towards the tube, the precursor vapor used for this study, i.e.  $\text{SiCl}_4$  vapor, is channeled through the cleaned and surface heated tubes. Hence, it is crucial to heat the tube evenly so as to achieve uniform axial deposition. One thing to note is that the  $\text{SiCl}_4$  is highly volatile and expands in volume when changing states. It is also highly sensitive to moisture, and producing corrosive HCl fumes. Hence a cool and dry environment is a must for safety.

To fabricate the active fiber, rare-earth (RE) dopants need to be deposited on the inner side of the hollow tube. Conventionally, this is done either by

solution doping (Townsend et al., 1987) or nanoparticle deposition (Tammela et al., 2002). We employed the former method for our mini preform fabrication for its simplicity.

The RE dopant solutions are prepared by mixing and dissolving various weight ratios of RE chloride hydrates in methanol. In this investigation, the following RE solutions were used: 10 g of  $\text{YbCl}_3 \cdot 6\text{H}_2\text{O}$  and 20 g of  $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$  dissolved in 200 ml of methanol for Ytterbium doped fiber, and 1 g of  $\text{ErCl}_3 \cdot 6\text{H}_2\text{O}$  and 20 g of  $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$  dissolved in 200ml of methanol for Erbium doped fiber. High RE doping concentrations were selected to demonstrate the feasibility of the proposed technique.

The vapor-deposited tubes were then immersed in the RE dopant solutions for 30 to 60 minutes, blow-dried by oxygen gas and heated at about 200 °C for 5 minutes to evaporate residual solvent and moisture. We then replicate the recipe-driven fire polishing, sintering, collapsing and sealing processes in the three-electrode plasma system. The process is semi-automated using National Instruments LabVIEW software to improve its repeatability and efficiency. Instead of moving the flame as in the standard fabrication process, we move the tube back and forth within the stationary ‘Ring of Fire’. Depending on factors such as thickness of deposited soot and RE layers, tube dimensions, and material properties of the tube, the process requires optimization of variables (e.g. plasma power, tube traversing speed) for good performance as well as the elimination of trapped air bubbles in the preform. For ~ 20 mm long preform used in this study, the entire sintering, collapsing and sealing processes took about 30 – 40 minutes. The average collapsed tube diameter was ~ 870  $\mu\text{m}$ . The typical recipe used for fabrication, and its corresponding outer diameter is depicted in Figure 2.

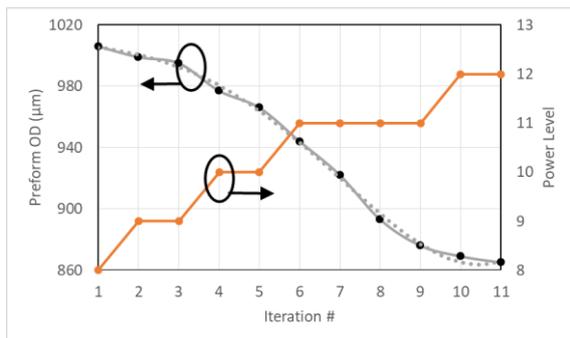


Figure 2: Typical recipe used in the process and its corresponding outer diameter.

In standard fiber pulling process, the preform is heated close to the melting point of silica in a furnace

or oven at the top of the fiber drawing tower, a thin fiber can then be pulled from the lower end of the preform. The intended fiber diameter can be controlled by the pulling speed and the furnace temperature. Before the fiber is wound up, it usually receives a polymer coating such as acrylate, silicone or polyimide for mechanical and chemical protection.

Since our fabricated preforms are significantly smaller, the above mentioned method is not feasible. Here, the horizontal tapering method was adopted instead. Similarly, the pulling speed and the power of the flame require optimization for desired fiber diameters.

In principle, the fabricated preforms can be drawn into fibers of any diameter narrower than that of the preforms. The maximum fiber length that can be drawn from a given preform dimension is calculated based on the following relationship, where  $l_p$  is the length of preform,  $r_p$  and  $r_f$  are the radii of the preform and fiber respectively.

$$l_f = l_p \left( \frac{r_p}{r_f} \right)^2 \quad (1)$$

### 3 RESULTS AND DISCUSSION

#### 3.1 Physical Dimensions

Using the proposed method, we have successfully fabricated several ~ 20 mm long ~ 870  $\mu\text{m}$  diameter preforms doped with either Ytterbium or Erbium. These preforms were pulled to fibers with a final diameter of 400  $\mu\text{m}$  and lengths ranging from 50 to 75 mm. The typical fiber end face of the fiber is depicted in Figure 3. The core is clearly seen, albeit not perfectly circular in shape. This is mainly due to the unevenness in the solution doping or slight misalignment of the tube in the ‘Ring of Fire’. As we pointed out at the beginning of this article, the focus of our proposed technique is rapid prototyping at early research phases, prior to scale-up and / or further development for applications. The typical cladding size of the fibers is shown in Figure 4. The maximum cladding size variation is estimated to be  $\pm 10 \mu\text{m}$ , or about  $\pm 2.5 \%$  for the 400  $\mu\text{m}$  fiber. The refractive index profiles of the fabricated fibers vary from one to another, depending on the solution composition and recipe used in the development. Nonetheless, a typical index profile is depicted in Figure 5 for illustration purpose.

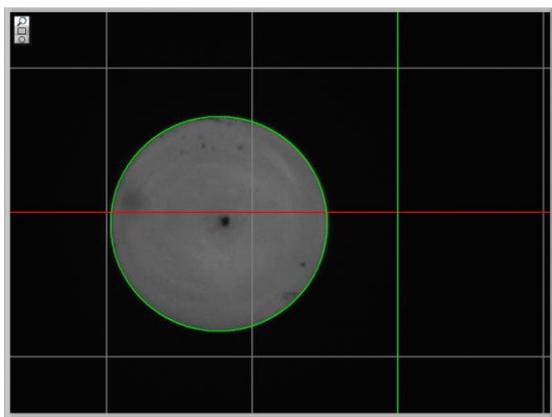


Figure 3: Typical fiber end face.

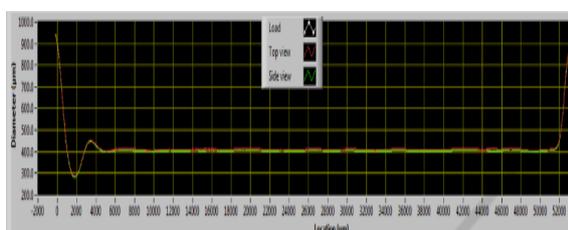


Figure 4: Cladding diameter showing uniformity throughout the axial length.

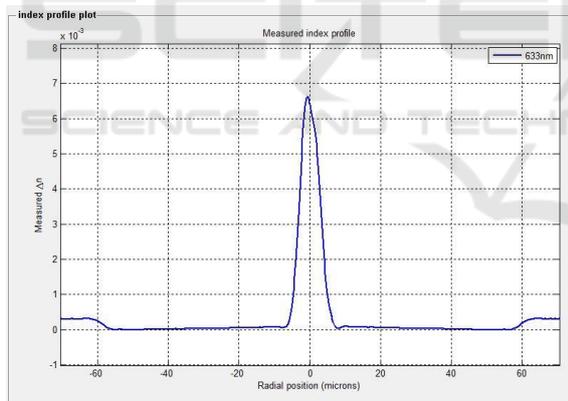


Figure 5: Typical refractive index profile of the fabricated fiber.

### 3.2 Spectral Performance

To analyze the spectra behavior of the fabricated fiber, the fiber was spliced to a piece of passive fiber for ease of connection and analysis. It was illuminated by a tungsten halogen light source covering the spectral range from 360 nm to 2000 nm, and pumped either by 915 nm or 976 nm laser sources, depending on the fibers' RE dopants. The spectra were captured using the optical spectrum analyzer (OSA) AQ-6315.

Reference spectrum was also taken using the similar manner, however with only passive fiber.

The spectra behavior of the fabricated ~ 50 mm Ytterbium doped silica fiber when pumped at 915 nm and 976 nm are illustrated in Figure 6 (a) and (b). The emission spectra of the fiber (with respect to the reference) can be clearly seen even for such a short length of fiber. We believe that this is mainly due to the high doping concentration of the RE element, which in turn demonstrated the capability of our fabrication technique and its potential with other RE dopant choices. More importantly, this marks the initial success of the method.

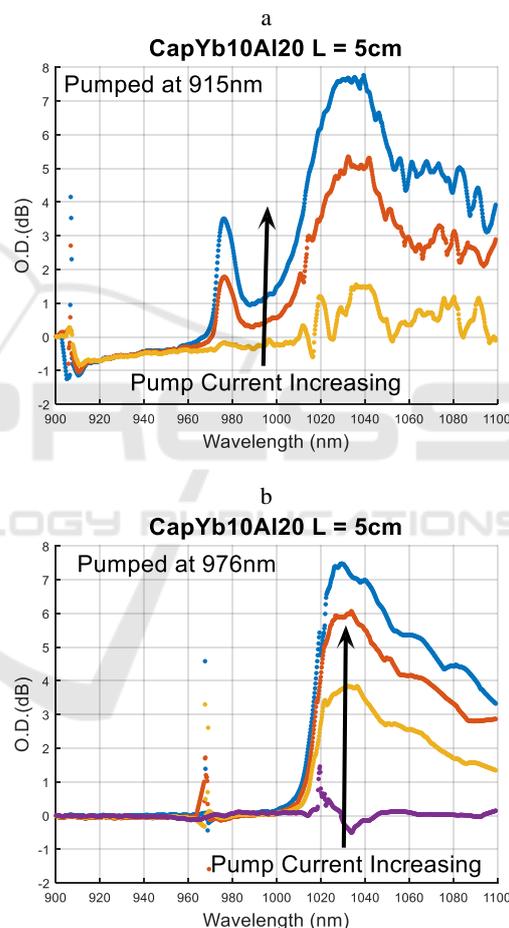


Figure 6: Transmission spectra of the fabricated Ytterbium doped silica fiber when pumped at (a) 915 nm; (b) 976 nm.

We further investigate the method using Erbium as another active element. The spectra of the fiber when pumped at 976 nm were obtained as follows. Once again the amplified spontaneous emission (ASE) spectra of the Erbium doped silica fiber can be observed, indicating the successful incorporation of Erbium dopants within the fiber.

Although the fibers fabricated show promising spectral results, they suffer from high background loss, this is mainly due to the purity and preparation environment and method of the samples. Furthermore, clean room environment was not utilized during the process.

Nonetheless, we would like to perceive this shortcoming in a positive manner: i.e. clean room environment is not necessary for this prototyping process, although it would be good for potential better results. To further improve the fabrication precision, tighter control of the temperature is essential. This “quick and dirty” process can be adopted until one is satisfied with the doping solution composition before switching it to the standard fabrication for good quality fibers.

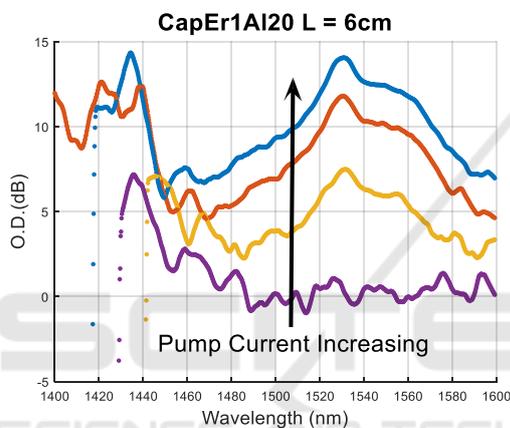


Figure 7: Spectra behavior of the fabricated Erbium doped silica fiber when pumped at 976 nm.

## 4 CONCLUSIONS

In conclusion, we have demonstrated a concept of miniaturizing the standard fiber fabrication processes to a benchtop version with repeatable results. We have also successfully fabricated rare earth doped, i.e. Ytterbium and Erbium doped silica fibers using the method incorporating with solution doping technique, and obtained reasonable spectra results. Undeniably, the length and quality of the fibers are traded-off by the time and cost involved. This process is beneficial for those who are requiring small quantity of the specialty optical fiber for rapid prototyping purposes, especially for the less mature optical fiber technologies, such as soft-glasses fibers. It also opens up the possibilities of exploring other glass hosts with various materials and compositions in a smaller scale.

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