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# PROCESS DEVELOPMENTS AND APPLICATIONS AROUND THE POWDER IN TUBE TECHNIQUE

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### Abstract

The purpose of this presentation is to give an overview of techniques available to produce original optical fibers and application dedicated. Some alternative fabrication solutions to the wellknow stack and draw technique will be developed, following by the interest to join the stack and draw technique to other ones like Repusil® Technique, Rod in Tube and finally the Powder in Tube methods, first described by the Bell Lab in the seventies and up to date since some years by several groups in the world. Realization of performing optical devices such as light sources or optical sensors can be purposed by this two last original ways.

## Introduction

Until the end of the nineties, the most common way to obtain preforms was the MCVD process, technique leading to optical fibres with very low attenuation losses mainly developed to telecommunication applications. In 1996, Bath University re-adapted an old technique using a stack of capillaries to obtain a structured preform [1]. This preform is drawn and provides the famous microstructured fiber or Photonic Crystal Fiber (PCF). Besides to give a new way to produce original structures, the technique opens new field applications to scientists, no longer limited to the telecommunication domain, where attenuation losses of the fiber were not so important. Thus, in parallel of these technical developments to provide very efficient optical fibers, some groups has begun to work on the fiber material, to propose that we call 'multi-material fibers', where core and cladding could be in different material compositions. J. Ballato's from Clemson University, P. Russel's from Erlangen in Germany, have developed some solutions with pure Germanium or Silicium core in order to mix electronic and optical behaviours. Since some years we developed the process of the

'powder in tube' to produce optical fibres where core is made with an original glass composition.

# Fabrication Processes to produce multimaterial fibres

## Rod in Tube method

The Rod-in-Tube method is a well-established technology for the fabrication of step index preforms and optical fibres. Due to the separate preparation of the core and cladding a wider range of material compositions and even almost non-compatible glass combinations are possible. One drawback is the additional interface between core and cladding, which can cause scattering with implying additional losses. Therefore a careful preparation (grinding, polishing, cleaning) of surfaces is necessary.

A core rod has to be stacked in a geometrical adjusted cladding tube. Two ways of further fabrication are possible: i) the core / clad materials will be fused together on a MCVD lathe and then drawn into a fibre or ii) if the expansion coefficient of core and cladding material is too different or the material is not be processed in a furnace or on the MCVD lathe, the core / cladding stack will be drawn under vacuum and melted together during fibre drawing.

## Repusil® Technique

The producible quantity overcomes by far the geometrical limitations of MCVD [2]. An other technique to produce high purity optical preforms has been developed by the IPHT (Jena, Germany) and Heraeus Quarzglass Company. It is based on the sintering of fused silica powders to produce very homogeneous rare earth doped silica rods used as the fiber core material.

An aqueous suspension of very pure  $SiO_2$  particles is doped in a similar way to the solution technique of MCVD layers [3]. The dopants were added as a mixed solution of suitable compounds of Aluminum and Ytterbium. The main difference in comparison with the MCVD technique is that the  $SiO_2$  particles are homogenously dispersed in a liquid suspension and are not directly deposited within a tube. This procedure is favorable to get enough material for green body forming with typical weights of 10 to 100 g.

After some additional processing and purification steps the processed doped granulates were sintered into homogeneous and bubble free Yb-doped bulk silica rods with diameters up to 30 mm without higher radial and axial doping level variations or fluctuations of the refractive index level. For high power application up to 4 kW this technology has been proven [4,5].

This technology can be expanded to other rare earth dopants like Thulium or Lanthanum to enhance possible applications like Tm based lasers ( $\lambda > 1900$  nm) or SC generation based on higher material nonlinearity

## Powder in Tube Method

We find some information and developments of this technique in the seventies, work done by the Bell Lab when scientists tried to find the best way to obtain fibers with low attenuation losses [6]. Unfortunately this technique couldn't compete with a technique developed at the same time, the MCVD process. More recently, SILITEC Fibre SA, patented an original route to produce industrial preforms, using silica sand [7] to produce the cladding of the fibers. Collaboration between SILITEC and Xlim gave us the possibility to adapt and develop this technique to produce core and/or cladding of the fibre with granulated glasses. Some fibers with cordierite material (mineral magnesoaluminosilicate glass) have first demonstrated in 2008 the capability of this technique to produce fibres with moderate attenuation losses (some dB/m in the visible range) compatible with the targeted applications requiring only some meters of fibres [8] (figure 1). This technique can be joined with the Stack and Draw Process to obtain original PCF (figure 3).



Figure 1: First optical fiber with cordierite core (10dB/m) - 2008



Figure 2: Silica doped with lanthanum oxide core (1.5dB/m) -



Figure 3: A PCF fiber where silica core is replaced by a Lanthanum oxide doped silica glass - 2011

Of course attenuation losses of fibres realised using this technique are far from ones given by the MCVD Process. One main question is to determine where these losses are coming from. To answer to this question, we realised optical fibres with wellknown material like N-SF6 glass from Schott Company, where bulk attenuation losses are known [9]. The N-SF6 in core/silica in cladding step index fibre presented attenuation losses closed to the one of the bulk material [10]. That means that the process does not add optical losses.

The use of granulated materials can involve porosity and specific heat treatment must be first done on the glass powder (around the  $T_G$ ) and the preform before the drawing process (high temperature vitrification step) to avoid any defect into the fibre core such as air bubbles. This work has been done in collaboration with Silitec Fibre SA in order to develop a specific set up allowing a vitrification of the granulated glass before drawing the preform (figure 4).



Figure 4: Vitrification Unit



Figure 5: Refractive index profile of the lanthano-aluminosilicate based fibre

By this way we are able to produce preforms without any structural default such as bubbles, and then suitable optical fibres for specific applications. As an example, the Figure 5 shows the refractive index profile of a  $SiO_2-Al_2O_3-La_2O_3$  core fibre obtained thanks to a strong collaboration with IPHT (Jena). Such optical fibres can have attenuation losses around 0,5 dB/m at 1550 nm [8] allowing efficient pumping at this wavelength and exacerbating non linear effects to realize SC sources.

Moreover, this original process allows the realization of non-standard optical fibres, with noncircular shape for example. Original glass material can then be inserted anywhere in the preform and finally, it is possible now to combine more than two materials in the same preform.

We have done also a theoretical work on such fibre structures. Due to the material behaviour and very high refractive index values, modelizations have shown that a high  $\Delta n$  step-index optical fibre can present a zero-dispersion wavelength (ZDW) as low as the one of a PCF whose core diameter is quite the same. Moreover, models also predicted that a high  $\Delta n$  step-index optical fiber which a core diameter of 5 µm can present a non linear coefficient ( $\gamma$  in W<sup>-1</sup>.km<sup>-1</sup>) as high as the one of a air/silica microstructured fiber with silica core diameter of 1 µm. By using this process, a step index fibre has been produced to give an original supercontinuum source [8].

## **Application Domains**

Optical source domain is very interested by the multimaterial optical fibres; we can change the usual materials to modify the linear and non-linear response of the guide, include rare earth-elements for amplification, or some other elements, like metallic particles, to enhance some non-linear responses.

Another promising way for these original fibres is sensing applications. Indeed, by introducing metallic particles inside the glass matrix, we modify one physical parameter of the glass: the thermal expansion coefficient, which can provide to the fibre, an extreme sensitive answer, giving an interesting route to discriminate thermal and mechanical behaviour of fibre sensors.

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