

# Tellurite photonic crystal fiber made by a stack-and-draw technique

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## Abstract

We report a method for producing, from the raw materials, high optical and geometrical quality glass tubes and photonic crystal fiber (PCF) preforms, without using extrusion or drilling at any stage. A thermal glass study was carried out in order to choose the appropriate glass composition to avoid crystallization problems during the tube, preform and fiber fabrication. A two period PCF was fabricated in addition to a co-doped Erbium and Thulium photonic crystal fiber. In the latter, a 187 nm wide amplified spontaneous emission (ASE) spectrum was obtained when pumping a 15 cm long fiber at a wavelength of 790 nm.

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## 1. Introduction

Photonic crystal fibers (PCF) are a new kind of waveguide, an optical fiber with high or low refractive index insertions running along all its length [1,2]. In these structures light can be trapped in the XY plane (perpendicular to the propagation direction) by means of two main physical mechanisms—total internal reflection or photonic band gap [3]. While the former resembles the guiding mechanism of standard fibers, in the PCF case no chemical doping is necessary. The effective refractive index difference between the core and the surrounding region is obtained by ‘doping’ the cladding with micrometer-size low index material insertions, being air holes in the most usual case. The second possible way to trap light in a PCF core is by means of coherent scattering from the high index parts of the cladding. In this case the light is guided in a low refractive index core [4] with even air guiding being possible [3].

As for standard fibers, PCF’s are also made in two main stages: manufacturing the preform and drawing it to fiber.

There are several techniques to prepare the microstructured preform depending on its material (silica, non-silica glasses, polymer etc). While for soft materials (such as polymers) the holes can be directly drilled in the preform [5], for silica the most convenient way is to prepare the preform by hand. The technique consists in stacking capillaries and rods, thus forming, on a macroscopic scale, the desired fiber microscopic geometry [1,2]. This procedure is usually called the ‘stack-and-draw technique’ and is used when it is possible to obtain high quality (both optically and geometrically) tubes.

For glass made ‘in-house’ directly from the raw materials the more standard procedure is to prepare the preform by extrusion, in which the melted glass is forced against a dye with proper geometry. Extruded photonic crystal fibers have already been prepared in the last three years with tellurite [6], SF6 [7] and SF57 [8] glasses. Contamination problems, the difficulty of preparing fibers with different glasses and/or complex geometries, and the importance of properly designing the dye are just some of the drawbacks of this technique.

To overcome these problems, it is proposed in this paper, for the first time, to extend the stack-and-draw technique to ‘homemade’ glasses, particularly to

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tellurite-based ones. Starting from the raw materials, high quality tellurite tubes are prepared (not extruded) and subsequently drawn to capillaries and rods. These are then stacked to form the desired preform, which is finally pulled to fiber.

Previous work used extrusion or drilling [9] to prepare the tubes from bulk (non-silica) samples, thus strongly reducing the inherent advantages of the stack-and-draw process.

The ability to prepare high quality tubes from ‘home-made’ glass starting from the raw materials expands the already extremely wide range of possibilities created by PCF’s.

In this work we discuss the tellurite-based glass composition used to minimize crystallization problems obtained via glass differential thermal analysis (DTA), and also the procedure used to prepare the tubes. A two-period PCF will be shown as an example of this powerful fabrication technique. Furthermore, to fully take advantage of the whole process, an erbium and thulium co-doped fiber (intended for optical amplification applications around 1550 nm band) will also be shown together with its amplified spontaneous emission (ASE) spectrum.

## 2. Glass composition and DTA measurements

The  $\text{TeO}_2\text{-WO}_3\text{-Na}_2\text{O-Nb}_2\text{O}_5$  composition was used, which is different from conventional tellurite glass in that this has two glass-forming compounds— $\text{TeO}_2$  and  $\text{WO}_3$ . This implies a more complex structure with a greater quantity of dopant sites or, in other words, the emission bandwidth enhancement [10]. To study some glass compositions based on this same set of compounds, looking for the best candidate to form tubes (and ultimately fibers), bulk samples were prepared. The raw materials (40 g in total) were melted in a platinum crucible for 30 min at 850 °C.

While the general behavior of the glass transition temperature ( $T_g$ ) of a binary glass can be predicted ( $T_g$  increases with  $\text{WO}_3$  content and reduces with  $\text{Na}_2\text{O}$  content [11,12]), to do a full investigation of such a complex glass a DTA characterization is necessary. The samples were milled and DTA measurements were taken for 50 mg of powder glass with grains between 60 and 150  $\mu\text{m}$  in size, placed within alumina crucibles and subjected to a heating rate of 10 °C/min. From these measurements it is possible to obtain the temperatures of glass transition, onset crystallization ( $T_x$ ) and melting ( $T_m$ ).

The results of the DTA measurements for different samples are shown in Fig. 1 and the respective  $T_g$ ,  $T_x$  and  $T_m$  are indicated. Glasses presenting a high thermal stability ( $T_x - T_g$ ) and a low temperature interval ( $T_m - T_x$ ) are the best candidates for tube/fiber fabrication due to the correspondingly small chance of crystallizations problems. The relevant parameter that encapsulates both of these characteristics is the Hruby Number ( $H_R$ ) defined as  $(T_x - T_g) / (T_m - T_x)$  [13]. A summary of the characteristic temperatures

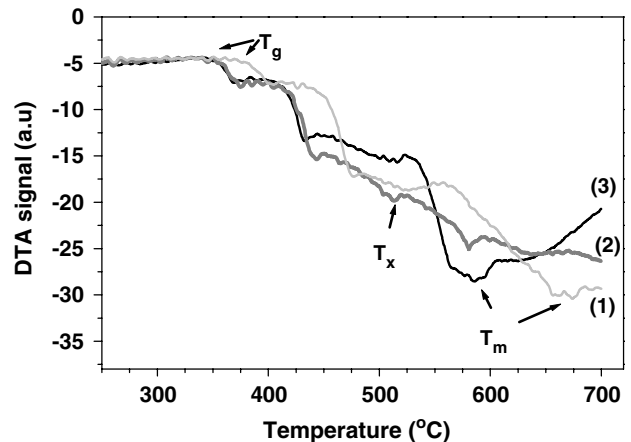


Fig. 1. Differential thermal analysis of three different tellurite glasses (1, 2 and 3 from Table 1). Characteristics temperatures, transition ( $T_g$ ), onset crystallization ( $T_x$ ) and melting ( $T_m$ ), are indicated.

Table 1

Glass samples with different compositions, characteristic temperatures and Hruby numbers

Sample	Composition (%mol)	$T_g$ (°C)	$T_x$ (°C)	$T_m$ (°C)	$H_R$
(1)	66TeO <sub>2</sub> –18WO <sub>3</sub> –7Na <sub>2</sub> O–9Nb <sub>2</sub> O <sub>5</sub>	380	526	674	0.98
(2)	70TeO <sub>2</sub> –19WO <sub>3</sub> –7Na <sub>2</sub> O–4Nb <sub>2</sub> O <sub>5</sub>	353	513	580	2.38
(3)	71TeO <sub>2</sub> –22.5WO <sub>3</sub> –5Na <sub>2</sub> O–1.5Nb <sub>2</sub> O <sub>5</sub>	352	517	585	2.42

of three different glasses is shown in Table 1 (corresponding to the curves in Fig. 1) together with their Hruby numbers.

It was found empirically that tellurite glasses with  $H_R > 2.3$  are good candidates for tube fabrication via the centrifugation method (see next section) while for  $H_R < 1.0$  crystallization problems occur during the process. By this approach, the composition was chosen to be 71TeO<sub>2</sub>–22.5WO<sub>3</sub>–5Na<sub>2</sub>O–1.5Nb<sub>2</sub>O<sub>5</sub> (%mol).

## 3. Results and discussion: tubes and preform fabrication

The tube fabrication process used in this work was centrifugation. Firstly, the melted glass (in a platinum crucible) is sucked inside a silica tube by means of a vacuum pump – and this quickly solidifies as a hollow tube or a solid rod. In both cases it presents low optical quality, with the presence of frozen bubbles. In the case of a hollow tube, it also gives a varying internal diameter. The second stage consists in holding the silica tube in a drilling chuck. A flame torch with planar and uniform temperature distribution is slowly, and precisely, positioned close to the silica tube, which is then heated while rotating at high speed.

The initial formation process of a tellurite glass tube is illustrated in Fig. 2, for a temperature above  $T_g$  and a low centrifugation speed ( $\sim 750$  rpm). It is possible to see (arrow) the internal hole being formed – please note two horizontal lines on the left-hand side of the tube. After the process is initiated, the rotation speed is increased ( $\sim 1800$  rpm) in order

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