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**Regular Articles** 

# Optical aging observation in suspended core tellurite microstructured fibers under atmospheric conditions



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## ABSTRACT

Tellurite glasses are good candidates for the development of broadband supercontinuum (SC) laser sources in the  $1-5 \mu m$  range. At the moment, beside very few exceptions, SC generation in TeO<sub>2</sub>-based microstructured optical fibers (MOFs) is limited to 3  $\mu m$  in the mid-infrared (MIR). We present here an observation of an optical aging occurring in six-hole suspended-core tellurite MOFs. When exposed to atmospheric conditions, such fibers show an alteration of their transmission between 3 and 4  $\mu m$ . This aging phenomenon leads to the growth of strong additional losses in this wavelengths range over time. Impact of the transmission degradation on spectral broadening is studied through numerical simulations of SC generation.

## 1. Introduction

The major revolution of microstructured optical fibers (MOFs) [1] in nonlinear optics has triggered the development of new optical functions and fiber-based small size systems. Most of early work was achieved on silica materials, but during the last decade, other vitreous matrices gained interest. Fluoride, as well as chalcogenide and heavy oxide glasses were considered in order to fill in silica's lack in terms of transparency window towards longer wavelengths [2]. Thereby, investigation of compact optical devices operating further in the infrared (IR) was possible. In light of those previous studies, we have worked on the development of compact mid-IR supercontinuum-based laser sources using suspended-core tellurite fibers. This kind of waveguide is very interesting since it allows the management of group-velocity dispersion, the control of the zero dispersion wavelength (ZDW) location, as well as higher nonlinear properties through the tight confinement of the guided modes in a small core surrounded by air-holes. Combined with the strong nonlinear nature of TeO2-based glasses and their wide transparency window (from visible to 6 µm on bulk), tellurite MOFs are therefore highly suitable for supercontinuum generation (SCG) in the mid-IR.

The use of vitreous materials for applied systems shows a great potential but caution must be taken, especially in terms of stability and durability. More particularly, glasses can exhibit chemical aging. Those changes are generally induced by external reagents and mainly depend on the glass environment. Previous studies [3,4] have shown that

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http://dx.doi.org/10.1016/j.yofte.2017.09.005 Received 23 January 2017; Accepted 3 September 2017 1068-5200/ © 2017 Elsevier Inc. All rights reserved. chalcogenide materials are concerned by this aging process and see their optical and mechanical properties change over time when exposed to moisture. According to [5,6] it is also true for fluoride glasses, well known for being sensitive to moisture and which strength as well as optical properties degrade in water containing environments. These observations imply that, when used for applied systems, those glasses must be protected from moisture in order to ensure the proper functioning of the devices over time. In the case of all-solid optical fibers, such protection is simple to achieve as polymer coating can be applied during the drawing process. For MOFs however, the situation is different. Their architecture, consisting of a lattice of parallel air capillaries along a glassy central rod, represents a source of contamination by diffusion of atmospheric moisture through the holes of the microstructure [7,8]. Airproofing such micrometric size systems can be a real challenge, as unlike the coating, it cannot be automated, and is hard to achieve without compromising light coupling into or decoupling from the fiber. Another solution would be to use the entire fiber device in a dry controlled atmosphere. In recent studies [7,8], we recorded the aging process occurring in suspended-core chalcogenide fibers, then confirmed by others [9]. We observed that only 24 h of exposure to ambient atmosphere (50% humidity, 24 °C) are enough to completely compromise the quality of our As<sub>2</sub>S<sub>3</sub> glass fibers. Absorption peaks related to hydroxyl groups or hydrogen bonding (O-H, As-OH and S-H) appear and grow over time preventing the use of our MOFs for SCG at full potential. A comparable phenomenon was observed in microstructured polymer optical fibers (MPOF) [10]. Moisture stream present in the air diffuses inside a six-hole fiber, thus leading to the distortion of the microstructure, formation of defects, and compromising the waveguide properties. Silica MOFs are also prone to an aging process [11]. A time-dependent growth of OH-related absorption peaks centered at 1364 nm, 1384 nm, 1398 nm and 1900 nm was observed in silica MOFs and hollow core bandgap fibers. All those studies point out the aging process inherent to MOFs attributed to the ingress of contaminants, atmospheric steam most of the time, in the microstructure.

In a previous work [12] we reported the generation of a 2000 nm bandwidth supercontinuum in a fluoro-tellurite suspended core fiber. The glass used for the MOF manufacturing had been purified from water by the means of zinc fluoride. OH concentration as low as 1 ppm was reached, allowing transmission of light up to 4  $\mu$ m on multimode single-index fibers. Despite those improvements in terms of material purity, the spectral broadening did not occur further than 2.8  $\mu$ m in small-core MOFs. This result did not match with the numerical calculations which predicted SCG up to 4  $\mu$ m. Additional losses had to be taken into account for the simulations to match the experimental results. The same kind of observation was made on chalcogenide MOFs and further investigations revealed the optical aging of our As<sub>2</sub>S<sub>3</sub> suspended core fibers, due to a contamination from atmospheric steam [7]. We therefore suspected that the same kind of phenomenon occurred in our TeO<sub>2</sub> based MOFs.

Thus, in this work, we report the time-dependent evolution of the optical properties of tellurite microstructured fibers exposed to ambient atmosphere. For that purpose, the evolution of the transmission of sixhole suspended core MOFs was recorded during several days.

#### 2. Experiments

#### 2.1. MOFs samples preparation

The glass rods dedicated to the manufacturing of low-OH MOFs were fabricated by the conventional melt-quenching technique, starting from high purity raw materials. The mixed batches were melted in a platinum crucible at 850-900 °C, during 2 h. In order to avoid the tellurium oxide reduction, the glass synthesis was performed under an oxygen gas flow (3 L/min, [H<sub>2</sub>O] < 0.5 ppm vol.) in a furnace mounted on a water-free glove box under dry air  $([H_2O] < 1 \text{ ppm vol.})$ . This system allows the stirring of the melts in an OH-free atmosphere, for the oxidation and homogenization of the glass without the risk of contamination from hydroxyl compounds. The hot liquid mixes were quenched into a brass mold preheated 100 °C under the vitreous transition temperature (Tg) and subsequently annealed at Tg for 8 h and finally slowly cooled down to room temperature.

Typically, 60 mm long glass rods with a 16 mm outer diameter were obtained from 60 g starting batches. Three MOFs of the following glass compositions (mol.%) were investigated in this work:  $80 \text{ TeO}_2 - 10 \text{ ZnO} - 10 \text{ Na}_2\text{O}$  (TZN), 75 TeO<sub>2</sub> - 15 ZnO - 5 ZnF<sub>2</sub> - 5 Na<sub>2</sub>O (TZNF) and 73 TeO<sub>2</sub> - 14.5 WO<sub>3</sub> - 10 ZnF<sub>2</sub> - 2.5 Nb<sub>2</sub>O<sub>5</sub> (TWNbF). The mechanical drilling technique was used to prepare the preforms which

were then drawn into six-hole suspended core MOFs (Fig. 1). The fibers have a 200–230  $\mu$ m outer diameter and a diameter core ranging from 8 to 14  $\mu$ m.

#### 2.2. Optical loss measurements

The optical losses of single-index large-diameter fibers were estimated using the cutback method on several meter long samples. The different spectra were recorded by the means of a Fourier Transformed Infrared (FTIR) spectrometer (NICOLET 6700) between 1 and 5 µm.

#### 2.3. Infrared spectroscopy

The IR transmission spectra of 60 cm long suspended core MOFs samples exposed to room atmosphere were recorded between 1  $\mu$ m and 4.2  $\mu$ m for 7–10 days. During the experiments, the temperature varied from 20 to 26 °C and the relative humidity (HR) was around 50%. Measurements were carried out by the mean of a NICOLET 6700 FTIR spectrometer using an external halogen lamp, emitting in the 0.1–5  $\mu$ m range. The light was injected into the core of the MOFs with a reflective objective in order to avoid chromatic effects. The guided signal was then focused onto a cooled InSb detector using an optimal aspheric AMTIR lens. The fiber samples was mounted onto 3 axis holders at both ends in order to optimize the coupling and detection conditions. Additionally, an infrared camera was used at the output end of the MOFs to control that the light was exclusively guided in the core, to avoid any fiber cladding contribution.

#### 3. Results and discussion

The transmission spectra for TZN, TZNF and TWNbF bulk samples and the respective attenuation spectra measured on several meter-long single-index fibers are shown in Fig. 2a and b. Usually, even if the glass is synthesized under dry atmosphere, the TZN fibers does not transmit light further than 2.8  $\mu$ m [13] because of strong water absorptions at 3.4 and 4.4  $\mu$ m. Nevertheless,

OH-groups content can be reduced in those materials using halogenide dehydration reagents like NaCl, NaF,  $ZnF_2$  and others [14–17], allowing light transmission on fiber further than 3  $\mu$ m.

To this aim, a zinc fluoride containing composition TZNF was developed, with a fiber transmission extended to 4.2  $\mu$ m as seen in Fig. 2b. Moreover, the TWNbF glass was studied for its particularly high potential for the manufacturing of low OH glasses. It is sodium free, an element known for its propensity to reduce glass durability, and contains an important amount of tungsten, which is not favorable to the presence of water in the vitreous matrix [18]. However, WO<sub>3</sub> and Nb<sub>2</sub>O<sub>5</sub> possess higher phonon energies (respectively 925 and 680 cm<sup>-1</sup>) compared to TeO<sub>2</sub> (650 cm<sup>-1</sup>) [19]. The presence of tungsten and niobium oxides in the glass matrix shifts the multiphonon edge to shorter wavelengths [16] as seen in Fig. 2a. Therefore, WO<sub>3</sub> and Nb<sub>2</sub>O<sub>5</sub> based glass fibers see their transmission limited to 3.5  $\mu$ m, as shown in Fig. 2b. Both TZNF and TWNbF glasses show a low water-



Fig. 1. SEM cross section pictures of (a) TZNF, (b) TWNbF and (c) TZN MOFs.

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