# Linear and nonlinear optical properties of chalcogenide microstructured optical fibers

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## ABSTRACT

Chalcogenide glasses are known for their large transparency in the mid-infrared and their high linear refractive index (>2). They present also a high non-linear coefficient ( $n_2$ ), 100 to 1000 times larger than for silica, depending on the composition. we have developed a casting method to prepare the microstructured chalcogenide preform. This method allows optical losses as low as 0.4 dB/m at 1.55 µm and less than 0.05 dB/m in the mid IR. Various chalcogenide MOFs operating in the IR range has been fabricated in order to associate the high non-linear properties of these glasses and the original MOF properties. For example, small core fibers have been drawn to enhance the non linearities for telecom applications such as signal regeneration and generation of supercontinuum sources. On another hand, in the 3-12 µm window, single mode fibers and exposed core fibers have been realized for Gaussian beams propagation and sensors applications respectively.

Keywords: Chalcogenide glass, microstructured optical fiber, Infrared, Wavelength conversion

#### 1. INTRODUCTION

Compared to oxide based glasses, vitreous materials composed with chalcogen elements (S, Se, Te) can present large transparency windows in the infrared. Indeed, chalcogenide glasses can be transparent from the visible up to 12-15  $\mu$ m, depending on their compositions. In addition, chalcogenide glasses contain large polarisable atoms and external lone electron pairs which induce exceptional non linear properties. Consequently, the non linear properties can be 100 or 1000 times as high as the non linearity of silica [1-3]. The manufacturing of small-core fibers (diameter smaller than 5  $\mu$ m) can be of great interest to enhance the non linear optical properties for telecom applications such as signal regeneration [4, 5], conversion to the mid infrared using Raman shifting [6, 7] and generation of supercontinuum [8-12].

An original way to obtain single-mode fibers is to design microstructured optical fibers (MOFs). In addition, these fibers present unique optical properties thanks to the high degree of freedom for designing the geometrical structure. After the first realisation of silica suspended core MOF in 1973 by Kaiser and his colleagues [13], and mainly after the work of T.A. Birks, P. Russel and J. Knight in 90's [14], this new class of fibers has attracted much interest. In the last 10 years, MOFs were realized with chalcogenide glasses. Obtained in 2000, the first holey chalcogenide fiber did not show any light guidance [15]. Since then, chalcogenide MOF with light guidance [16]. The common method to prepare MOF is the stack-and-draw technique. This method is widely used for silica MOF [14]. In 2008, it has been shown that optical losses in chalcogenide fibers were due essentially to the presence of scattering defects at the interface between capillaries [17]. So, to avoid interfaces defects, MOFs have been prepared by a newly established casting method [18]. Thanks to this technique, chalcogenide MOFs with low optical losses have been elaborated [7].

In this paper, the realization of low losses and innovative chalcogenide MOFs such as mid-IR endlessly single fibers, allsolid fibers, small core and exposed core fibers will be described. The small core fibers were used for wavelength conversion by nonlinear effects and an exposed core fiber was used for the detection of infrared signatures of organic molecules.

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#### 2. ELABORATION OF THE MICROSTUCTURED OPTICAL FIBERS

A classical method to make MOFs is the stack and draw technique [14, 19]. However, that technique induces high optical losses (several dB/m) in chalcogenide glass MOF, due to the poor quality of interfaces between capillaries [17]. Then, we have developed a new casting method to fabricate chalcogenide performs [18]. For MOF, glass samples with the composition  $As_{38}Se_{62}$  and  $Ge_{10}As_{22}Se_{68}$  are fabricated and purified by using the usual sealed silica tube method. The methodology for elaborating the fiber consists in molding a rather fluid liquid glass into a silica vessel which contains aligned silica capillaries. After controlling that the liquid has filled the empty space, the silica vessel is quenched to room temperature. This operation is followed by the dissolution of the silica capillaries by an HF treatment. The mould is entirely made of silica capillaries thread into silica hexagonal guides. The silica guides are prepared by slicing a silica microstructured perform [18]. After dissolution of silica the resulting microstructured chalcogenide preform is drawn into fiber under an He controlled atmosphere. Typically, the fibers diameter can vary from 100 to 300 µm. During the drawing step, the hole diameters are adjusted by applying a pressure into the preform holes. This method permits to obtain various geometries. For example, the fibers can have 3 rings of holes (Fig 1a). The core size can also be controlled, and it is possible to obtain a very small core, to exacerbate the non linear properties of the chalcogenide glass (Fig 1b, 1c). The optical losses of the fibers prepared by the molding process can be less than 1 dB/m at the telecom wavelength and can reach less than 0,1 dB/ in the mid-IR at 3.7 µm [7, 20].



Figure 1: Different geometries obtained with chalcogenide glasses, (a) three rings large core fiber [7] (b) small suspended core [7], (c) three rings small core fiber [20] and (d) exposed core fiber [21]

## 3. INFRARED TRANSMISSION AND OPTICAL LOSS

Before the elaboration of the microstructured optical fibers high quality chalcogenide glasses has been prepared in order to obtained low loss fibers. Thus, the glasses were synthesized from high purity elements by successively melting and quenching the glass melt in a sealed silica ampoule. The glasses were then purified thanks to dynamic and static distillations. These steps enable to remove both volatile and refractive impurities such as water, oxides, carbon etc. The purified batch was finally placed in a rocking furnace for several hours, quenched and annealed at the glass vitreous transition temperature. Optical attenuations of the fibers have been measured with a Bruker FTIR by the classical cut back technique. The black body light of the FTIR is injected into the fiber and the signal is detected with a nitrogencooled MCT (mercury, cadmium, tellurium) detector. Due to the large size of the injected spot, the light is not injected only in the core of the fiber. Indeed, part of the IR light is injected in the clad of the fibers and propagates in the clad. In order to detect only the light that propagates in the core, a high-refractive-index Ga-Sn alloy is applied on the surface of the fiber in order to remove the cladding modes. Indeed, the Ga-Sn alloy presents a very weak reflection coefficient together with high loss which leads to an absorption of the cladding light after only few cm. The attenuation curve of the glass used (composition  $As_{38}Se_{62}$ ) for the realization of the microstuctured optical fiber is given in figure 2. The figure 2 presents also the attenuation of two different microstructured optical fibers fabricated with the same glass  $As_{38}Se_{62}$ . Thus, the attenuation curves of the two microstructured fibers can be compared with the material once. The first MOFs, is a large core (24 µm) multimode fiber, and the second one is an endlessly single mode fiber with a core diameter of 14 μm. The optical losses are slightly more important in the microstructured fibers, more particularly for the shorter wavelengths. These additional optical losses are a combination of the intrinsic guiding losses due to the geometry of the

fiber and extrinsic losses due to the presence of additional scattering defects. One can note that the signal-to-noise ratio is poor for the single mode fiber. This is due to the small size of the core and a smaller numerical aperture of this fiber. Indeed, the power intensity from the black body of the FTIR injected in the core of the fiber is lower in the case of the single mode fiber. It can be notice that the optical losses is around/or less than 1dB/m from 2 to 9.5  $\mu$ m for all the fibers. An absorption band, due to the presence of Se-H chemical bonds, reaches 3-4 dB/ m at 4.55  $\mu$ m. However, this transmission window corresponds to the molecular fingerprint region where molecules have strong absorption bands. This glass composition is consequently good candidate for broadband single mode guiding and chemical sensing.



Figure 2 . Attenuation of  $As_{38}Se_{62}$  fibers, (a) single index fiber (glass wire), (b) endlessly single mode microstructured optical fiber, (c) large core microstructured optical fiber

#### 4. ALL-SOLID ALL-CHALCOGENIDE MICROSTRUCTURED OPTICAL FIBER

Light propagation by modified total inter reflection can be obtained in a microstuctured fiber by substituted the air inclusions by other low index inclusions. As example, air inclusions can be filled by another chalcogenide glasses with a lower refractive index than the  $As_{38}Se_{62}$ . So, a  $As_{40}S_{60}$  / $As_{38}Se_{62}$ . The preform was elaborated by a three-step process. First, the As-Se glass rod was molded on a homemade 3 rings of silica capillaries mold has described in [18]. The outer diameter of the resulting preform is 20 mm with holes diameter above 500 µm. Secondly, the  $As_{40}S_{60}$  rod was drawn directly into a 480 µm diameter fiber and the obtained fiber was cut into 6 cm long pieces. Finally, these red As-S sticks have been thread into the holes of the As-Se preform. The MOF is obtained by drawing the two-glass preform (Fig 3). Spaces between the As-S sticks and the As-Se preform were eliminated upon drawing thanks to a vacuum system. The low refractive index glass matrix is  $As_{40}S_{60}$ , with indices varying between 2.48 and 2.38 in the mid-infrared. The high-index inclusions are made with  $As_{38}Se_{60}$  glass, whose refractive index is in the range of 2.82–2.75, depending on the infrared wavelength.

To observe the light guidance a monochromatic light ( $\lambda$ = 1.55 µm) guided in a single mode silica fiber was butt-coupled with a 15 cm long chalcogenide MOF. The image of the fiber output was visualized on an IR camera (Fig. 3b). In spite of several injection conditions, no modification of the quasi-gaussian mode profile was observed. The optical losses of fibers have been measured on FTIR apparatus with a cooled MCT detector (Fig. 3c). The optical losses of the As-S/As-

Se MOF were determined while a GaSn alloy was applied on the surface of the fiber to inhibit cladding mode guidance (Fig. 3b, 3c). One can note that the near field measurements indicate a single mode propagation. Numerical simulations have shown that the fundamental mode exhibits less than 1dB/m in the 1-8  $\mu$ m wavelength range while the guiding losses of the second mode range from 100 dB/m at 1  $\mu$ m and more than 1000 dB/m at 8  $\mu$ m [22]. Consequently, from a practical point of view the fabricated fiber can be considered as single mode in the whole wavelength range 1-8 $\mu$ m because after few centimeters it is not possible to detect a second mode with such high losses.



Figure 3 : all-solid fiber with low index inclusion . (a) Cross section (b) Single mode propagation at the telecom wavelength (1.55  $\mu$ m, (c) optical loss.

A second all solid chalcogenide MOF has been elaborated by substituted the low index inclusions based on the As-S glass by high index includions based on a Te-As-Se glass. In this fiber, The low refractive index glass matrix is  $As_{38}Se_{62}$ , with indices varying between 2.82 and 2.75 in the mid-infrared. The high-index inclusions are made with  $Te_{20}As_{30}Se_{50}$  glass, whose refractive index is in the range of 2.96–2.90, depending on the wavelength. Such configuration is also known under the acronym ARROW for Anti Resonant Reflecting Optical Waveguides [23] even if, in this case the emphasis is given to the property of the individual scatterers with high refractive index and not to the collective effect building the bandgap [24, 25]. The parameters of the fibers presented in figure 4 are the following : outer diameter 230  $\mu$ m, d/ $\Lambda$  is equal to 0.374 while the inclusion diameter is 5.5  $\mu$ m.



Figure 4 : All-solid fiber with high index inclusion : (a) cross section, (b,c) near field measurements propagation at 3.39  $\mu$ m, (d) Transmission bands (arbitrary units)

The transmission band of the fiber has been also mesured by using a FTIR spectrometer. In this case the attenuation of the fibers was two high to obtained the optical losses of the fiber by the classical cut back technique. However, transmision band of the fiber can be measuraed in arbitrary units (Fig. 4). The shape of the transmission curve and the presence of the transmission bands is typically the signature of a photonic band gap propagation. One transmission band is centered arround 3.3  $\mu$ m. In order to qualify the ligth propagation in a such fiber, near field measurement at 3.39  $\mu$ m have been achieved. Due to the low power of the laser and significant optical losses of the core of the fiber, near-field measurements have been carried out for a 4 cm-long fiber. Indeed, the experimental optical losses of the core were estimated to be in the 20 – 50 dB/m range. Fig. 4 shows the near-field image obtained in a 4 cm-long sample of the band-gap fiber. The first near-field capture (Fig. 4b) is observed when the light propagates in the core and the clad of the fiber. The second near-field image (Fig. 4c) is obtained when a Ga-Sn alloy is applied at the surface of the fiber in order to remove the cladding modes. Numerical simulations taking into account the geometry and the material properties confirm the existence and the positions of the bandgaps, and also core guidance at the wavelengths utilized for the experiments [26].

#### 5. INFRARED SPECTROSCOPY

The chalcogenide fibers have been successfully implemented in fiber evanescent spectroscopy experiments, for detection of biochemical molecules in various fields of applications including water pollution [27], microbiology and medicine[28]. It has been shown that, the infrared signature of gaz embedded in the holes of chalcogenide MTIR-MOF can be detected. Indeed, in reference [29], the signature of CO<sub>2</sub> gaz has been observed at 4,2  $\mu$ m, through a 36 holes As<sub>2</sub>Se<sub>3</sub> MOF filled by pure CO<sub>2</sub>. However, in this case, the design does not permit a real contact between the core of the fiber and the chemical species. Indeed, to be detected, the chemical species must fill the fiber holes. Besides, a new configuration of microstructured fibers has been developed: microstructured exposed-core fibers [30]. This design, which was proposed for the first time by Y.L. Hoo and al. in 2003, consists of an optical fiber with a suspended micron-scale core that is partially exposed to the external environment [31]. In ref [21], the demonstration of an exposed core chalcogenide MOF has been demonstrated. This configuration has been chosen to elaborate a chalcogenide fiber for chemical species detection. The figure 5 gives infrared signatures of propan-2-ol molecules recorded with a 110  $\mu$ m diameter single index fiber and a chalcogenide MTIR-MOF presenting a core diameter of 15  $\mu$ m (with an outer diameter of 220  $\mu$ m). Although evanescent wave absorption is inversely proportional to the fiber diameter, the result shows that an exposed-core fiber is much more sensitive than a single index fiber having a twice smaller external diameter.



Figure 5 : Infrared signature of Propan-2-ol on the surface of a single index fiber (outer diameter =  $110 \mu$ m) and a chalcogenide exposed core MOF (outer diameter =  $220\mu$ m, core diameter =  $15\mu$ m).

# 6. NON LINEAR PROPERTIES: WAVELENGTH CONVERSION AND SUPERCONTINUUM GENERATION

Another remarkable property of chalcogenide glasses is their strong optical non linearity. Indeed, the nonlinear index of selenium-based glasses can be more than 1000 times larger than that of silica. This property is an important requirement for nonlinear applications such as wavelength conversion. In addition, small-core fiber geometries (diameter smaller than 5  $\mu$ m) can enhance dramatically the intrinsic nonlinear optical properties of chalcogenide fibres. In order to observe strong nonlinear effects, small core fibers were elaborated (Fig 1b, 1c) and studied at the telecom wavelength and in mid-infrared. At the telecom wavelength, low threshold ( < 6 mW) Brillouin laser was obtained in a small core single mode fiber Fig 1c[18] and all optical wavelength conversion and time domain demultiplexing with a 170 Gb/s rate were demonstrated [19] . Farther in infrared, by pumping at 2  $\mu$ m, four-cascaded Raman shift [32] and supercontinuum between 1.5 to 2.8  $\mu$ m [33] were observed in a suspended core fiber (in nanosecond and picosecond regime respectively). By pumping at 3.7  $\mu$ m (OPO fs source), in one of our As-Se suspended core, a broadband supercontinuum has been obtained between 1.5 to 8  $\mu$ m [34]. The average power at the output of the chalcogenide MOF was 16 mW. The length of the fiber was 18 cm and the core diameter was about 4  $\mu$ m.

# 7. CONCLUSION

Since the first realization in of a chalcogenide microstructured optical fiber [15], strong improvements have been obtained: regular geometries, low losses fibers, single mode fibers and highly nonlinear fibers. These improvements open new investigations in numerous filed of application such as, telecom functions, interferometry, generation of new infrared sources and sensors.

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