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## Chalcogenide microstructured optical fibres for mid-IR applications

*Fibres optiques microstructurées en verres de chalcogénures pour l'infrarouge moyen*Johann Trolès<sup>a</sup>, Laurent Brilland<sup>b</sup><sup>a</sup> Équipe Verres et Céramiques, ISCR, UMR CNRS 6226, Université Rennes-1, 35042 Rennes cedex, France<sup>b</sup> SelenOptics, 263, avenue du Général-Leclerc, 35042 Rennes, France

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## ABSTRACT

Compared to oxide-based glasses, vitreous materials composed of chalcogen elements (S, Se, Te) show large transparency windows in the infrared. Indeed, chalcogenide glasses can be transparent from the visible up to 12–18  $\mu\text{m}$ , depending on their compositions. In addition, chalcogenide glasses contain large polarisable atoms and external lone electron pairs that induce exceptional non-linear properties. Consequently, the non-linear properties can be 100 or 1000 times as high as the non-linearity of silica. An original way to obtain single-mode fibres is to design microstructured optical fibres (MOFs). These fibres present unique optical properties thanks to the high degree of freedom in the design of their geometrical structure. Various chalcogenide MOFs operating in the IR range have been elaborated in order to associate the high non-linear properties of these glasses with the original MOF properties. Indeed, chalcogenide MOFs might lead to new devices with unique optical properties in the mid-infrared domain, like multimode or endlessly single-mode transmission of light, small or large mode area fibres, highly birefringent fibres and non-linear properties for wavelength conversion or generation of supercontinuum sources.

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## R É S U M É

Les verres de chalcogénures sont des verres à base d'éléments chalcogènes (S, Se et/ou Te). Ces verres présentent une large fenêtre de transparence dans l'infrarouge. En effet, les verres de chalcogénures peuvent être transparents du visible jusqu'à 12–18  $\mu\text{m}$ , suivant leur composition. De plus, les verres de chalcogénures présentent des atomes fortement polarisables (atomes lourds avec la présence de paires électroniques libres), qui induisent de fortes propriétés optiques non linéaires. Par exemple, l'indice de réfraction non linéaire peut être de 100 à 1000 fois supérieur à celui des verres classiques à base de silice. Les fibres dites microstructurées permettent, grâce à la grande diversité de leur géométrie, d'obtenir des propriétés originales de propagation de la lumière. Dans le présent article, différentes géométries de fibres microstructurées en verres de chalcogénures seront présentées. Ces fibres peuvent être monomodes, quelle que soit la longueur d'onde, et présenter de très petites tailles de mode ou, au contraire, un très large mode de propagation. Les fibres peuvent également être très fortement biréfringentes et peuvent

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être utilisées pour faire de la conversion de longueur d'onde et obtenir ainsi de nouvelles sources laser à fibre, très rares dans l'infrarouge moyen.

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## 1. Introduction

Compared to oxide-based glasses, vitreous materials composed of chalcogen elements (S, Se, Te) present large transparency windows in the infrared. Indeed, chalcogenide glasses can be transparent from the visible up to 12–15  $\mu\text{m}$ , depending on their compositions. In addition, chalcogenide glasses contain large polarisable atoms and external lone electron pairs that induce exceptional non-linear properties. Consequently, the non-linear properties can be 100 or 1000 times as high as the non-linearity of silica [1,2]. The manufacturing of small-core fibres (diameter smaller than 5  $\mu\text{m}$ ) can be of great interest to enhance the non-linear optical properties for telecom applications such as signal regeneration [3], generation of supercontinuum, and conversion to the mid-infrared using Raman shifting [4–7]. In optical transmission systems, non-linear effects in optical fibres can be used to realize all-optical processing such as data regeneration by self-phase modulation (SPM) or wavelength conversion by four-wave mixing (FWM).

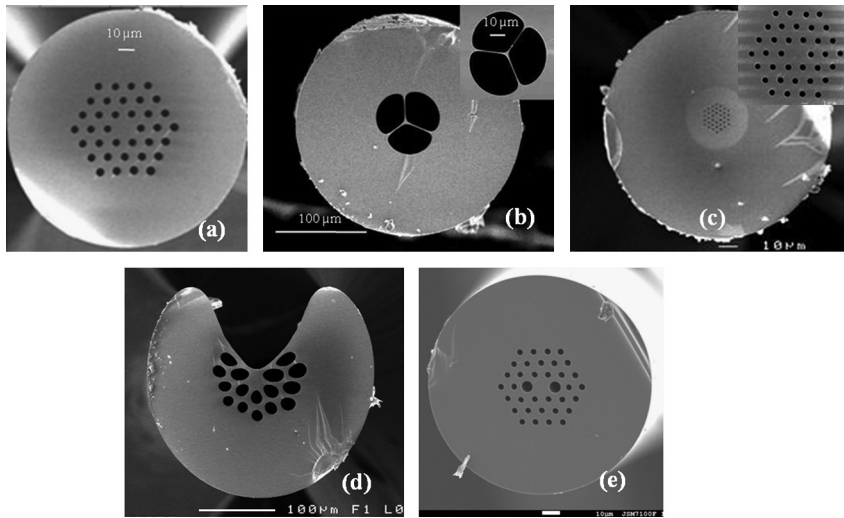
An original way to obtain single-mode fibres is to design microstructured optical fibres (MOFs). In addition, these fibres present unique optical properties thanks to the high degree of freedom for designing the geometrical structure. After the first realisation of a silica suspended core MOF in 1973 by Kaiser and his colleagues, and mainly after the work of T.A. Birks, P. Russel and J. Knight in 1990s [8], this new class of fibres has attracted much interest. In the last 10 years, MOFs were realized with chalcogenide glasses. Obtained in 2000, the first holey chalcogenide fibre did not show any light guidance [9]. Since then, chalcogenide MOF with light guidance was obtained in sulphur and selenide glassy systems [10,11]. The common method to prepare MOF is the stack-and-draw technique. This method is widely used for silica MOF [8]. In 2008, it has been shown that optical losses in chalcogenide fibres were due essentially to the presence of scattering defects at the interface between capillaries [12]. So, to avoid interfaces defects, MOFs have been prepared by a newly established casting method [11]. Thanks to this technique, chalcogenide MOFs with low optical losses have been elaborated [6]. The first chalcogenide MOFs exhibited a non-linear parameter  $\gamma$  ( $\gamma = 2\pi n_2/\lambda A_{\text{eff}}$ ) between 100 and 1500  $\text{W}^{-1} \text{km}^{-1}$  and optical losses at the telecom wavelength of 10–15 dB/m [10,13,14]. More recently, the non-linear parameters in a chalcogenide MOF reached values from 15,000 to 31,000  $\text{W}^{-1} \text{km}^{-1}$  with optical losses less than 1 dB/m [15,16].

In this paper, the realization of low losses and innovative chalcogenide MOFs such as small-core or exposed core fibres will be described and several examples of applications will be exposed. The small core fibres were used for wavelength conversion by non-linear effects: four wavemixing, Raman and Brillouin scattering and supercontinuum generation. Secondly, infrared detection obtained in the exposed core fibre will be described. And finally, an asymmetric fibre exhibiting high birefringence has been realized in order to obtain polarization maintaining in the 3–8- $\mu\text{m}$  region.

## 2. Elaboration of the microstructured optical fibres

A classical method to make MOFs is the stack-and-draw technique. However, that technique induces high optical losses (several dB/m) in chalcogenide glass MOF, due to the poor quality of interfaces between capillaries [12]. Then, we have developed a new casting method to fabricate chalcogenide performs [11]. For MOF, glass samples with the composition  $\text{As}_{38}\text{Se}_{62}$  and  $\text{Ge}_{10}\text{As}_{22}\text{Se}_{68}$  are fabricated and purified by using the usual sealed silica tube method. The methodology for elaborating the fibre consists in moulding a rather fluid liquid glass into a silica vessel that 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 [11]. After dissolution of silica, the resulting microstructured chalcogenide preform is drawn into fibre under a He-controlled atmosphere. Typically, the diameter of the fibres can vary from 100 to 300  $\mu\text{m}$ . During the drawing step, the diameters of the holes are adjusted by applying a pressure into the preformed holes.

This method permits to obtain various geometries. For example, the fibres can have three 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 (Figs. 1b, 1c). The optical losses of the fibres prepared by the moulding process can be less than 1 dB/m at the telecom wavelength, and can reach less than 0,1 dB/m in the mid-IR at 3.7  $\mu\text{m}$  [6,11].



**Fig. 1.** Different geometries obtained with chalcogenide glasses: (a) three-ring large-core fibre [6], (b) small suspended core [6], (c) three-ring small-core fibre [26], (d) exposed core fibre [27], and (e) highly birefringent fibre [25].

### 3. Wavelength conversion

One of the remarkable properties 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 fibre geometries (diameter smaller than 5  $\mu\text{m}$ ) can enhance dramatically the intrinsic nonlinear optical properties of chalcogenide fibres. In order to observe strong nonlinear effects, small-core fibres were elaborated (Figs. 1b, 1c) and studied at the telecom wavelength and in the mid-infrared window. At the telecom wavelength, a low threshold (<6 mW) Brillouin laser was obtained [17] and all optical wavelength conversion and time domain demultiplexing with a 170 Gb/s rate were demonstrated [16]. Farther into the infrared, by pumping at 4.4  $\mu\text{m}$  with a peak power of 5.2 kW, a supercontinuum from 1.7 to 8  $\mu\text{m}$  with an average output power of 15.6 mW has been obtained [18].

### 4. Infrared detection

One of the most specific characteristics of chalcogenide glasses and fibres is their IR transparency. One can note that the infrared window corresponds to the molecular fingerprint region where the chemical compounds have strong absorption bands. For infrared deputed spectroscopy, optical fibres define practical sensing tools. Indeed, they transport light to and from the sensing region. In such configurations, the interaction between the light and the environment occurs at the surface of the fibre. For that matter, in the past decade, chalcogenide glass fibres have been successfully implemented in evanescent wave spectroscopy experiments, for the detection of bio-chemical species [19,20]. In our study, we have developed an original design where the core of the fibre is exposed to the external environment (Fig. 1d). Organic compounds were put in contact with a 2-cm-long part of the fibre and the result has been compared with the ones obtained thanks to single index fibres having different diameters. Thus we have demonstrated that, when the light is confined in the 15- $\mu\text{m}$  core of the exposed-core fibre, the latter is much more sensitive than a single index fibre having a twice smaller diameter. As an example, Fig. 2 presents the detection of acetone IR signature obtained using an exposed core fibre.

### 5. Mid-IR polarization maintaining optical fibre

Microstructured optical fibres (MOFs) provide new solutions for obtaining strong birefringence. Thus, high birefringence can be achieved by designing an asymmetric arrangement of air holes, where holes with two different diameters are placed along the orthogonal axes near the core region [21]. This modification of the fibre symmetry results in an increase in the effective refractive index difference between the two orthogonal polarization modes. An asymmetric structure can be obtained in MOFs not only by altering the air holes size near the core area [21], but also by changing the shape of air holes [22], or by introducing mechanical stresses as demonstrated in [23]. It has been shown that the birefringence of silica polarization-maintaining microstructured optical fibres (PM-MOFs) reaches values of the order of  $10^{-3}$ , which is one order of magnitude higher than that of PANDA or bow-tie silica fibres. In this work, a chalcogenide PM-MOF operating in the 3–8.5- $\mu\text{m}$  infrared window has been investigated. The obtained fibre has an outer diameter of 125  $\mu\text{m}$  and a central solid core of 9  $\mu\text{m}$ . Fig. 1e shows a scanning electron microscope image of the fibre. Holes surrounding the core have a pitch of 7.64  $\mu\text{m}$  and diameters of 3.46  $\mu\text{m}$  for the small holes and 6.43  $\mu\text{m}$  for the large ones. Light propagation is demonstrated

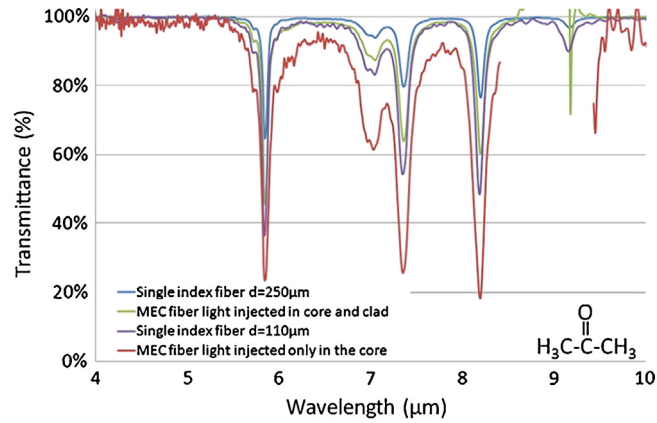


Fig. 2. Acetone detection with AsSe single-index fibre  $d = 250 \mu\text{m}$  (blue line),  $d = 110 \mu\text{m}$  (purple line) and with the exposed-core fibre, black body injected in core and clad (green line) and light signal in the core of the fibre only (red line).

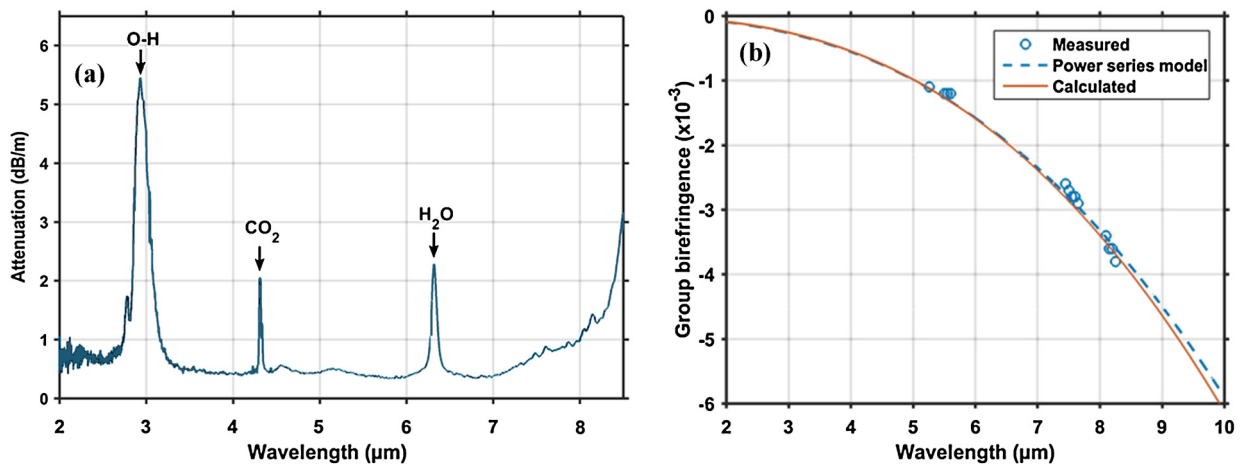


Fig. 3. Optical losses: (a) measured and calculated group birefringence; (b) loss of the polarization-maintaining fibre.

with injection of monochromatic light at  $3.39 \mu\text{m}$  from a He–Ne laser. The near-field image and 3D-profile at the output of the fibre are visualized with an infrared camera (not shown). The field distribution can be approximated by a Gaussian function in all directions, which indicates single-mode behaviour at  $3.39 \mu\text{m}$ . Consequently, single-mode propagation is expected within the transmission range of the fibre. The attenuation of the PM-MOF, determined by the standard cut-back technique, is shown in Fig. 2a. The attenuation curve presents extrinsic absorption bands at  $2.9 \mu\text{m}$ ,  $4.26 \mu\text{m}$ , and  $6.3 \mu\text{m}$  attributed to the presence of O–H groups, and of  $\text{CO}_2$  and  $\text{H}_2\text{O}$  molecules, respectively. Besides, the optical losses at  $1.55 \mu\text{m}$  are measured to be around  $0.9 \text{ dB/m}$ .

In this study, the group birefringence of the manufactured PM-MOF is determined by using the so-called crossed-polarizer technique [24,25]. The optical sources used for the measurement are transverse magnetic polarized Fabry–Pérot Quantum Cascade Lasers (FP-QCL) operating at  $5.25 \mu\text{m}$ ,  $5.55 \mu\text{m}$ ,  $7.55 \mu\text{m}$ , and  $8.2 \mu\text{m}$ . Computed and experimental values of birefringence are reported in Fig. 3b. Very good agreement between experimental and calculated values of birefringence is found.

## 6. Conclusions

The casting method is well adapted for the realisation of chalcogenide microstructured optical fibres. By this process, high-quality non-linear fibres have been prepared with optical losses smaller than  $1 \text{ dB/m}$ . Strong non-linear effects have been observed in such fibres and permitted to realize high-speed wavelength conversion and generation of supercontinuum in the mid-IR spectral range. On another hand, the infrared transmission of the chalcogenide fibres permits the realisation of infrared sensors. The high sensitivity of a microstructured fibre with an exposed core has been demonstrated by evanescent wave spectroscopy. In addition, a single-mode polarization-maintaining MOF covering the mid-IR region from  $3$  to  $8.5 \mu\text{m}$  has been obtained.

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