Mid-infrared 2000-nm bandwidth supercontinuum generation in suspended-core microstructured Sulfide and Tellurite optical fibers

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Abstract: In this work, we report the experimental observation of supercontinua generation in two kinds of suspended-core microstructured soft-glass optical fibers. Low loss, highly nonlinear, tellurite and As2S3 chalcogenide fibers have been fabricated and pumped close to their zero-dispersion wavelength in the femtosecond regime by means of an optical parametric oscillator pumped by a Ti:Sapphire laser. When coupled into the fibers, the femtosecond pulses result in 2000-nm bandwidth supercontinua reaching the Mid-Infrared region and extending from 750 nm to 2.8 µm in tellurite fibers and 1 µm to 3.2 µm in chalcogenide fibers, respectively.

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1. Introduction

Because of combined properties of high coherence, large bandwidth, brightness and potential compactness, supercontinuum generation has been a topic of high interest in nonlinear optics. Indeed, broadband light sources have found many applications in the field of spectroscopy, metrology, telecommunication or biology. To this aim, numerous efforts have been first dedicated to investigate supercontinuum generation in fused silica fibers with record brightness and spectral expansion ranging from ultraviolet (UV) to mid-infrared (MIR) [1–8]. However, since intrinsic transmission window of fused silica makes supercontinuum expansion a challenging task above 2.2 µm [9, 10], the recent trend is thus to provide alternative materials so as to spread further in the MIR region [11–26]. In this context, larger transmission windows and stronger nonlinear materials have been proposed as an alternative to the conventional fused silica such as tellurite [11–14], chalcogenide [15–20], heavy oxides [21, 22] or fluoride glasses [23–26]. Tellurite and chalcogenide glasses have serious advantages because of their wide transmittance window, chemical durability and high nonlinearity. Depending on their composition, infrared transparency can exceed 10 µm while the Kerr nonlinearity can be 500 times stronger than fused silica [27]. These different features make them serious candidates for broad mid-infrared supercontinuum generation. For example, supercontinuum as broad as 4000-nm bandwidth has been generated in a sub-cm long Tellurite microstructured fiber by Domachuk et al. in Ref [12], thanks to a femtosecond pulse pumping at telecommunication wavelength. Regarding chalcogenide fibers, pumping in the anomalous dispersive regime is a much challenging task since the chromatic dispersion contribution of the material generally induces a zero dispersion wavelength far in the infrared region (typically around 5 µm) and thus far from commercially available femtosecond pulse sources. Consequently, a careful design of the waveguide structure has to be performed so as to shift down the zero dispersion wavelength (ZDW) and thus allowing an anomalous dispersion pumping. To this aim, several strategies have been considered such as waveguide fabrication, tapering, nanowire and microstructured optical fibers, which give rise to efficient spectral broadening and supercontinuum generation [19, 20, 27–30]. In this work, we report the experimental fabrication and characterization of two kinds of soft-glasses, low loss, suspended-core microstructured optical fibers. More precisely, both tellurite and As2S3
chalcogenide suspended-core fibers with nearly 3-µm core diameter have been designed and pumped in their anomalous dispersion regime by means of an optical parametric oscillator delivering 200-fs pulses between 1700 and 2500 nm. When coupled into the fibers, the nJ-level femtosecond pulses result in 2000-nm bandwidth supercontinuum spreading in the mid-infrared region until 2.8 µm in Tellurite fiber and 3.2 µm in chalcogenide fiber.

2. Tellurite fiber design

The fiber preform is first elaborated from a tellurite 80TeO₂-10ZnO-10Na₂O glass (mol. %) synthesized by the fusion method under oxidizing atmosphere. High-purity, commercial raw materials are used for the glass fabrication without any additional purification: tellurium (IV) oxide (Alfa Aesar, 99.9995%), zinc oxide (Alfa Aesar, 99.999%) and sodium carbonate (Alfa Aesar, 99.997%). The oxide mixture is melted in a platinum crucible at a temperature of 800-850°C between 1h and 2h. The resulting melt is then poured in a brass mould preheated at 270°C. The annealing of the glass sample is finally completed around its transition temperature (Tg = 285°C) during several hours before being slowly cooled down to room temperature. The resulting glass rod is thus characterized by a typical length of 4.5 cm and a round diameter of 16 mm. The preform elaboration is achieved by means of a mechanical drilling process, which ensures better loss performances than the traditional time-consuming, numerous handy manipulations stack-and-draw technique [16, 31]. The resulting preform consists in a triangular solid core surrounded by three holes of typically 0.8-mm diameter and 30-mm length. In a final stage, the preform is then drawn to fiber under a helium atmosphere. Indeed, from glass classical DSC measurements at a heating rate of 10°C/min between room temperature and 400°C, no exothermic crystallization peak is registered. Thus, the temperature difference between Tg and crystallization temperature is higher than 115°C, making indeed the glass suitable for fiber drawing which is performed around 360°C.

The transmittance of the bulk was first measured using a FTIR Fourier Transform InfraRed spectrometer (Perkin–Elmer Spectrum One) on a 4-mm thick rod sample for two casting processes, more precisely under room atmosphere conditions and under dry atmosphere in a glove box. Figure 1 shows typical transmittance spectra recorded in the range of 1300-6700 nm.

![Fig. 1. Transmittance of the tellurite bulk as a function of wavelength measured on a 4-mm thick sample. Glass casted under room atmosphere (red solid line), glass casted under dry atmosphere in a glove box (blue solid line).](image)

The crucial point here is the residual OH absorption which induces excess of losses. Indeed, the tellurite glasses, as all oxide glasses, are known to suffer from a strong water absorption in the infrared range that decreases the glass transmission [31–33]. In the case of the glass elaborated under air atmosphere (red line in Fig. 1), we can clearly observe two main OH bands: strongly bonded OH (strong OH) and weakly bonded OH (weak OH) combined with free OH. This OH pollution is mainly due to reaction between the atmospheric moisture and the glass batch during glass synthesis. The central positions of these bands depend on the degree of OH hydrogen bonding (H-bonding) present in the glass [34]. The
strongly bonded OH absorb around 4350 nm, while the combination of weakly H-bonded OH and free OH in the glass provides a huge 1-µm bandwidth absorption band located around 3350 nm, which results in a poor 15% transmission at 3.3 µm and thus preventing any practical application beyond 3 µm. In order to overcome this issue, we have completed the glass fabrication process under a dry atmosphere inside a glove box. Resulting transmission spectrum is then depicted in Fig. 1 thanks to the blue solid line. We can notice a high flatness of the transmittance level around 76% until 2.8 µm as well as a significant reduction of the OH absorption band around 3.3 µm, for which transmittance is greatly improved from 15% to 70%.

More precisely, the concentration of OH-groups can be estimated by means of the following equation [35]:

$$N_{\text{OH}} = \frac{N_A}{\varepsilon \cdot L} \cdot \ln\left(\frac{1}{T}\right)$$

where $N_A$ is the Avogadro constant, $L$ the sample thickness (cm), $T$ the transmittance, and $\varepsilon$ the molar absorption of the free OH groups in the glass. In the calculations we use the molar absorption of the OH groups in silicate glasses (49.1x10$^3$ cm$^2$/mol) [36]. Then the amount of OH-groups can be presented in parts per million (ppm) [37]. After calculations, we found that the glass fabrication under dry atmosphere allows us to reduce the OH-ions concentration by a factor 36: 510 ppm under air atmosphere vs 14 ppm in the glove box. In this latter case the origin of the remaining OH-groups is attributed to the initial pollution of the raw materials.

After drawing process, we obtained the fiber profile illustrated in Fig. 2(a) captured by means of a scanning electron microscope (SEM). The resulting tellurite fiber is characterized by a 120-µm outside diameter with a typical suspended-core shape consisting in a triangular 3.4-µm solid core surrounded by three petal-like holes with 1 µm thickness bridges. Because of the critical small size of the microstructured fiber core, a measurement of fiber spectral losses could only be successfully performed on a large core quasi mono-index profile fiber, which is drawn just before the microstructured fiber itself, in the early stage of the drawing process, when the holes of the microstructure are not yet under pressure. It is thus possible to correctly inject the light coming from a Fourier Transformed InfraRed (FTIR) spectrometer in this large core fiber. The cut-back technique is implemented for that purpose. Typically, cut back measurements are performed on 5 meters of fiber, with around 20% accuracy, attributed to variations of the fibre cross section when cutting the fiber. Note additionally that fiber losses were also measured punctually at 1.55 µm for the small-core microstructured fiber itself, at a level of 1.5 dB/m, thus corroborating previous results on quasi mono-index one. Spectral fiber losses are nearly flat up to 2.5 µm but increase rapidly beyond 2.7 µm. This flatness was also confirmed experimentally by the resulting supercontinuum spectrum obtained in section 5. It is also important to notice here that, even if we achieved a strong reduction of the residual OH absorption in the bulk glass, the cumulative effect of these linear losses in the fiber remain dramatic for practical application above 2.8 µm. This point underlines the fact that an order of magnitude improvement is still required regarding the OH-groups concentration, so as to achieve a concentration below 1 ppm.
Group velocity dispersion of the tellurite fiber has been also measured thanks to the well-known low coherence interferometric method, which has been proved to be convenient to characterize short segments of optical fibers [27]. Experimental results are compared to numerical simulations of modal properties based on the fiber geometry derived from the SEM analysis (see Fig. 3). The good agreement confirms that our fiber exhibits a zero-dispersion wavelength (ZDW) close to 1660 nm while the bulk glass is characterized by a ZDW close to 2.2 µm. The suspended core profile has then successfully allowed us to manage with the chromatic dispersion and to shift the ZDW to shorter values more suitable for SC generation.

3. As$_2$S$_3$ chalcogenide fiber design

For sulfide fibers, first of all, the As$_2$S$_3$ glass is prepared from elemental high-purity 5N starting products: arsenic and sulphur. However, initial sulphur is generally polluted by water and carbon, while the metallic arsenic surface is often polluted by As oxide [38]. Thus, the starting products have to be purified from their respective impurities by thermal treatment under a dynamic vacuum (10^{-6} mbar). Sulphur and arsenic are thus dried by heating for a few hours at respectively 120°C and 290°C [39]. Arsenic is then mixed with sulphur and the batch is sealed under vacuum in a silica setup. A static distillation is performed which allows the separation of carbon while the mixture is collected in a silica ampoule. Finally, the silica ampoule is sealed under vacuum, placed in a rocking furnace and heated up to 700°C at a slow heating rate (1 – 2°C min$^{-1}$). The heating treatment allows the fusion and the reaction of the starting products. Obtained liquid batch is refined at high temperature for 10 to 12 hours. The melt is then quenched at room temperature. Resulting solid glass rod is then annealed at
its glass transition temperature $T_g$ ($T_g \approx 200 \, ^\circ C$) for 12 hours and slowly cooled down to room temperature. A thick glass rod (7-cm long, 16-mm in diameter) is finally obtained at the end of this stage. As in the previous case of tellurite glass, DSC measurements at a heating rate of $10 \, ^\circ C/\text{min}$ between room temperature and 400 °C do not exhibit any exothermic crystallization peak so that the glass can be drawn into fiber at a temperature around 270°C, at which no re-crystallization phenomenon takes place.

The technique used to elaborate the chalcogenide microstructured optical fiber was the same as the previous tellurite one. The elaboration of the preform was based on a mechanical machining of the rod. Our process presents the originality to be performed entirely mechanically with the help of mechanical tools only and without the need of any ultrasonic assistance. This mechanical drilling process has been largely used in order to prepare a variety of geometric profiles and to control the positions and diameters of the holes, inner surfaces and core surfaces by optimization of the drilling conditions [16, 39, 40]. We designed a preform with three holes of typically 0.8 mm diameter and 30 mm length placed in a triangular arrangement around a solid core. Such a preform was then drawn into fiber in our laboratory under optimized conditions, leading to a 3.2-µm suspended-core fiber with 0.6 µm thickness bridges, which transversal section can be observed in Fig. 4(a). More precisely, the drawing process has been performed in an inert and dry atmosphere, at a temperature of 270°C and at a drawing speed of 5 meters/min.

As in the previous study on tellurite fibers, linear losses were first characterized on the pre-drawing mono-index chalcogenide fiber sample by means of the FTIR spectrometer. Experimental data are depicted in Fig. 4(b) in the range of 1 to 4.5 µm (blue solid-line). Experimental measurements show relatively flat spectral losses in the range from 1.5 to 2.5 µm with a minimum below 1 dB/m around the pump wavelength at 2.3 µm.

The attenuation curve obtained for this glass (Fig. 4(b)) shows that the material losses contribution is quite similar to our previous results in [16] and [39]. We can also see narrow and large extrinsic absorption bands, centered at characteristic wavelengths which corresponds to residual OH and SH pollution of the glass. SH concentration ($\approx 2.5$ ppm) can be estimated from the spectrum referring to [41] for which extinction coefficient associated to the SH vibration at 4 µm is $2.5 \, \text{dB/m/ppm}$. Finally, note that results of Fig. 4(b) are well confirmed by loss measurements in the suspended-core fiber at fixed wavelengths (1.06 and 1.55 µm, see Triangles in Fig. 4(b)), using a corresponding fixed wavelength laser source and the cut-back technique.

As in the previous study on tellurite fibers described above, group velocity dispersion of the chalcogenide suspended-core fiber has been also characterized using the same interferometric set-up [27]. Experimental results are depicted in Fig. 5 and compared to numerical analysis of the modal properties. We clearly reveal a zero-dispersion wavelength
close to 2330 nm, which has to be compared to the ZDW of the bulky sulfide glass located around 5 µm.

Fig. 5. Chromatic dispersion curve of the suspended-core chalcogenide fiber. Circles: experimental data, solid-line: numerical modeling.

4. Experimental set-up

The experimental set-up depicted in Fig. 6 consists in an optical parametric oscillator (OPO) pumped by a Ti:Sapphire laser. The OPO delivers 200-fs pulses at a repetition rate of 80 MHz with an average power close to 450 mW. The idler output, tunable from 1.7 to 2.6 µm was used to pump both tellurite and chalcogenide fibers under test in the anomalous dispersion regime and close to their ZDW in order to efficiently generate supercontinua [6]. Fibers are cleaved by means of a scalpel blade and quality of the interfaces is carefully checked under microscope before mounting the sample onto a 3-axis holder. Pulses are then focused into the fiber under test by means of a 20X microscope objective. At the output of the fiber, resulting supercontinuum is collected by a fluoride ZBLAN fiber and spectrally characterized by means of either two Yokogawa optical spectrum analyzers covering 350 to 1200 nm and 1200 to 2400 nm respectively or a FTIR spectrometer in the range of 2400 to 4000 nm.

Fig. 6. Experimental set-up used to generate supercontinua in tellurite and chalcogenide suspended-core fibers. FUT: Fiber under test.

5. Experimental results and discussions

5.1 Tellurite fiber supercontinuum

Figure 7(a) shows the experimental results achieved by pumping a 40-cm long sample of our tellurite fiber. The pump wavelength is fixed to 1745 nm and resulting spectra have been recorded as a function of injected input power. The nonlinear index of the material is given by $n_2 \approx 3.8 \times 10^{-19} \text{ m}^2/\text{W}$ at 1550 nm [42], which implies a nonlinear Kerr coefficient $\gamma = 175 \text{ W}^{-1}\text{km}^{-1}$ (the numerical effective mode area is 7.8 µm$^2$). We clearly observe the well-known physical picture of supercontinuum generation, mainly driven by self-phase modulation, soliton fission and Raman based soliton self-frequency shift [6], which allow extending the resulting spectrum towards the mid-infrared up to 2.8 µm for an input pump power of 112
mW. This input pulse power corresponds to a soliton number close to \( N = 25 \) and a pulse energy of 1.5 nJ. The resulting 112-mW supercontinuum is characterized by a 2000-nm bandwidth, corresponding to almost 2 octaves and is remarkably flat (a 1900-nm span contained in a \(-20 \text{ dB range}\)). Here, the corresponding output power is \(-90\%\) of the input power. Note also that the depicted data above 2.8 \( \mu \text{m} \) in the 112-mW spectrum of Fig. 7(a) corresponds to the noise floor level of the FTIR spectrometer. The lower edge of the supercontinuum is mainly enlarged through phase-matched dispersive wave generation. Indeed, during propagation the frequency-shifted solitons can trap short-wavelength radiations and shift them to yet shorter wavelengths. The shortest wavelength generated in the supercontinuum is then determined by the maximum soliton shift towards the infrared. This interaction is characterized by a group-index matching between both spectral components and it can be determined by analyzing the shape of the group index curve of the tested fiber \[7\]. In Fig. 8 we plot the experimental supercontinuum edges for different output powers together with the group index curve obtained numerically for the fundamental mode. We clearly join the points on either edge by straight lines, thus confirming both the concept of group-index matching as well as chromatic dispersion modeling and measurements on the whole span of wavelengths. Moreover, this physical process allowed us to efficiently optimize the experimental supercontinuum generation in the tellurite fiber without any need to continuously monitor the resulting spectrum on the whole wavelength range but just by focusing on the shortest wavelength spreading.

![Fig. 7.](image-url)

Fig. 7. (a) Experimental recording of supercontinuum generation in a 40-cm long suspended-core Tellurite fiber as a function of pump power. (b) Corresponding numerical simulations.

Figure 7(b) presents the numerical simulation corresponding to these experimental data and obtained through the split-step Fourier-based solving of the generalized nonlinear Schrödinger equation \[6\] taking into account the full dispersion curve from Fig. 3, measured fiber losses as well as self-steepening and analytical model of the Raman gain spectrum \[42–44\]. The numerical results fit quite well the dynamics observed in the experiments and confirm that the spectral broadening is not mainly limited by the intrinsic linear losses for the optimized power and fiber length used here. However we cannot extend the supercontinuum bandwidth in the mid-Infrared region with a longer fiber due to the residual OH absorption which, even if it has been greatly reduced in the fabrication process under dry atmosphere, still prevents some application beyond 3 \( \mu \text{m} \).
5.2 Sulfide fiber supercontinuum

Figure 9(a) illustrates the experimental results obtained from a 45-mm-long segment of our suspended-core chalcogenide fiber. Figure 9(a) shows the resulting supercontinuum for a pump wavelength of 2300 nm and an injected power of 70 mW (4.6 kW peak power). Here, the output power is ~70% of the input power. The nonlinear index of the material is $n_2 \approx 2.8 \times 10^{-18}$ m$^2$/W at 1550 nm [45], which gives a nonlinear Kerr coefficient $\gamma = 1175$ W$^{-1}$/km$^{-1}$ (the effective mode area is 6.5 µm$^2$). The injected pulse corresponds to a high soliton number $N = 84$ and a pulse energy of 0.9 nJ. The chalcogenide fiber is here clearly pumped close to the zero-dispersion wavelength, in the anomalous dispersion regime, and is characterized by a strong and rapid self-phase modulation process followed by soliton fission in the earlier centimeters of fiber. In this case the fission occurs randomly due to the significant role of modulation instability when $N >> 20$ [6]. Subsequent propagation is then associated with a degradation of the coherence. The resulting supercontinuum extends from 1200 nm to 3200 nm in the ~20 dB range (excluding the absorption peak at 2.9 µm). Note however that large residual OH absorption bands are clearly visible around 2.9 µm, which implies to work with only short fiber lengths. This point underlines the fact that OH and SH absorption bands characterized on the pre-drawing mono-index fiber and depicted in Fig. 4(b) are undervalued by this measurement method and that the drawing process as well as resulting OH induced absorption time deterioration of the microstructure dramatically increased these extrinsic absorption losses. Nevertheless, the resulting spectrum also indicates that supercontinuum extension is performed in the early millimeters of the fiber, allowing the solitons to shift out the spectrum above the absorption band.

Figure 9(b) represents the numerical simulations corresponding to these experimental results. Thanks to a careful modeling of transmission losses and Raman gain [46] and using the full dispersion curve from Fig. 5, we have obtained a good agreement which confirms our experimental observations. However, in order to better fit the experimental data, particularly the SH and OH absorption bands, extra losses have to be taken into account around 2.9 µm and 4 µm, which confirms the under-evaluation of these absorption bands measured in Fig. 4(b) and an ageing of our non coated fibers under room atmosphere. This point is actually under study. Nevertheless, numerical simulations depicted in Fig. 9(c) and neglecting extra losses of absorption peaks reveal that the dynamic of supercontinuum generation could be...
extended until 5.5 µm, thus confirming the dramatic limitation imposed by OH- and SH-groups induced extra losses.

![Graphs](image)

**Fig. 9.** (a) Experimental recording of supercontinuum generation in a 45-mm long sample of suspended-core chalcogenide fiber for a pump power of 70 mW (b) Corresponding numerical simulation (c) Corresponding numerical simulation without OH- and SH- groups absorption peaks.

6. Conclusion

In summary, we report here the experimental generation of supercontinuum in two kinds of suspended-core microstructured soft-glass optical fibers, namely tellurite and sulfide fibers. Thanks to an optical parametric oscillator pumped by a Ti:Sapphire laser and delivering 200-fs pulses between 1700 and 2500 nm, we achieved efficient pumping of both fibers above their respective zero-dispersion wavelength in the anomalous dispersion regime. When coupled into the fibers, the femtosecond pulses result in 2000-nm bandwidth supercontinua reaching the mid-infrared region and extending from 750 nm to 2.8 µm in tellurite fibers and 1 µm to 3.2 µm in sulfide fibers for input pulse energy of ~1 nJ, respectively.

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