Towards integration of a liquid-filled fiber capillary for supercontinuum generation in the 1.2–2.4 μm range

S. Kedenburg,* T. Gissibl, T. Steinle, A. Steinmann, and H. Giessen

4th *Physics Institute and Research Center SCOPE, University of Stuttgart,*
70550 Stuttgart, Germany

**s.kedenburg@pi4.uni-stuttgart.de*

Abstract: We demonstrate supercontinuum generation in unspliced as well as in integrated CS_2-filled capillary fibers at different pump wavelengths of 1030 nm, 1510 nm, and 1685 nm. A novel method for splicing a liquid-filled capillary fiber to a standard single-mode optical fiber is presented. This method is based on mechanical splicing using a direct-laser written polymer ferrule using a femtosecond two-photon polymerization process. We maintain mostly single-mode operation despite the multi-mode capability of the liquid-filled capillaries. The generated supercontinua exhibit a spectral width of over 1200 nm and 1000 nm for core diameters of 5 μm and 10 μm, respectively. This is an increase of more than 50 percent compared to previously reported values in the literature due to improved dispersion properties of the capillaries.

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1. Introduction

Optical fibers confine light in a very small area over a long interaction length. For that reason fibers are especially suited for enhancement of nonlinear effects such as supercontinuum generation [1]. Broad mid-infrared supercontinuum sources are very important for research because they can excite vibrational absorption bands in the molecular fingerprint region (2.5-25 μm), which makes it possible to specifically identify molecules and functional groups [2]. To overcome the limitation of a standard silica fiber with moderate nonlinear refractive index and strong material absorption above 2.4 μm, fibers filled with highly nonlinear liquids have recently come into focus [3–8]. They offer striking possibilities in the field of optofluidics because of a 100 times larger nonlinear refractive index compared to fused silica [9] and a high transmission range up to 12 μm with few absorption peaks in the mid-IR spectral region [10], which is crucial for extending supercontinua far into the mid-IR.

In selectively liquid-filled photonic crystal fibers as well as in liquid-filled fiber capillaries the dispersion properties can be tailored accordingly, e.g., by changing the core size or using mixtures of different liquids. This degree of freedom in the liquid-filled fiber waveguide design offers interesting opportunities to promote supercontinuum generation.

To avoid direct free space coupling into the liquid core and make the handling more practical integrated devices based on fusion splicing [11, 12] and mechanical splicing [7] have been developed. The challenge is to create a stable splice of a standard single mode fiber to a capillary fiber while still being able to fill the liquid into the hollow core and to avoid air bubbles at the same time.

Kieu et al. [11] introduce a fusion splicing method where a standard fiber is cleaved with an angle which helps to form a small gap after the fusion splice for liquid access. This gap splicing method works well since the capillary supports only the fundamental mode which is not the case for our investigated capillary core diameters filled with CS_2. Hence, a large extent of higher order modes is excited which results in the absence of spectral broadening. Also the method is not highly reproducible due to the critical overlap between both fibers to form a strong joint but not collapsing the hollow core.

A different intriguing approach developed by Xiao et al. [7] is based on mechanical splicing a capillary fiber to a standard telecom fiber by using a large diameter tapered capillary as sleeve. In this case, difficulties can arise from trapped air that can penetrate into the liquid-filled core when both fibers are spliced together which leads to significant propagation losses.

Here, we present a novel approach for an integrated liquid-filled fiber device which is based on mechanical splicing with the help of femtosecond two-photon direct laser writing of a polymer ferrule. Our approach overcomes all the aforementioned difficulties. We write an outer sleeve ring onto a standard single-mode fiber to aid with the alignment. The design of the ferrule is constructed such that no air bubbles can penetrate into the liquid core. The splicing process is highly reproducible due to the high accuracy of direct laser writing. Furthermore, this method is also suited for generating supercontinuum in liquid-filled capillaries that are not exclusively single-mode by maintaining mostly the fundamental mode of the spliced single-mode fiber due to the accurate mutual alignment. The larger core diameter capillaries of 5 μm and 10 μm in our experiment are multi-mode but possess beneficial dispersion properties that are suited for generation of a broad supercontinuum. With our unspliced as well as our integrated capillaries we are able to selectively excite the fundamental mode which results in broad white light spectra that range from 1200 nm up to 2400 nm.

The structure of the paper is as follows: First, the linear properties of the unspliced liquid-filled capillaries and the general measurement setup for supercontinuum generation are shown. Second, the measurement results for the supercontinuum generation in the unspliced capillaries are presented. Afterwards, the fabrication process of the integrated liquid-filled device based on mechanical splicing is described, followed by the experimental supercontinua of the integrated capillaries.

2. Properties of liquid-filled fiber capillaries

In our experiment we use commercial available (Polymicro Inc.) fused silica capillary tubes with inner diameters of 5 μm and 10 μm filled with carbon disulfide (CS_2). We work with the liquid CS_2 because of its very high nonlinear refractive index on the order of $n_{2I} = 2.7 \times 10^{-18}\,\mathrm{m}^2/\mathrm{W}$ [9], its good transparency in the infrared [10], and its good guiding properties. Due to the higher linear refractive index of the core liquid CS_2 [13] compared to

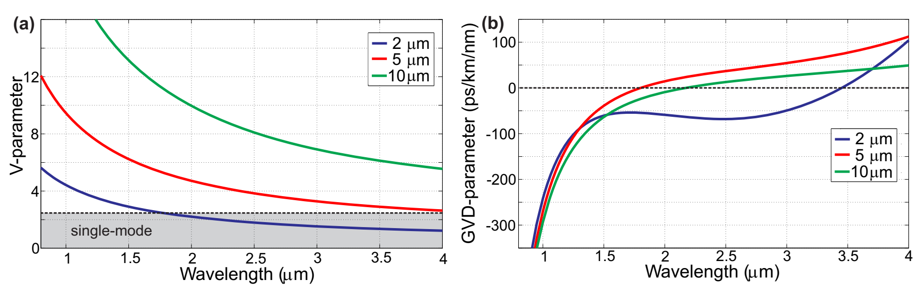

Fig. 1. (a) V-parameter for purely CS_2-filled capillary fibers for core diameters of 2 μm, 5 μm, and 10 μm. The single-mode boundary at V=2.405 is marked with a black dotted line. Only the 2 μm core capillary fulfills this requirement for pump wavelengths higher than 1.8 μm. For the 5 μm and 10 μm core diameters we have at all pump wavelengths a V-parameter that allows multi-mode propagation. (b) Group velocity dispersion (GVD) of the fundamental mode for purely CS_2-filled capillary fibers for core diameters of 2 μm, 5 μm, and 10 μm. The zero dispersion boundary is marked with a black dotted line. The zero-dispersion wavelength for the 5 μm core capillary is the smallest (1.8 μm) which is beneficial for supercontinuum generation. We operate at all pump wavelengths in the normal dispersion regime.

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the cladding material fused silica [14] the waveguiding is due to total internal reflection. The very high core-cladding index difference results in large values of the V-parameter (given by $V = 2\pi a/\lambda \sqrt{n_{\mathrm{core}}^2 - n_{\mathrm{clad}}^2}$ with a core radius) which is illustrated in Fig. 1(a). Both fiber core diameters will support multi-mode operation in our measured wavelength range. Only the 2 μm core diameter capillary fiber guides solely the fundamental mode ($V \leq 2.405$) for wavelengths higher than 1.8 μm. The drawback of purely single-mode fibers is that their mode confinement becomes worse for increasing wavelengths and more power is guided in the cladding which is not highly nonlinear. Another benefit of the 5 μm and 10 μm core diameter capillaries is their lower zero dispersion wavelength (ZDW) and hence a weak group velocity dispersion (GVD) parameter (given by $D = -\frac{2\pi c}{\lambda^2}\frac{d^2\beta}{d\omega^2}$) at pump wavelengths higher than 1.5 μm as displayed in Fig. 1(b). Generally, efficient and broadband SC-generation is obtained by pumping close to the ZDW of the fiber [1]. For this reason we focus our measurements onto the 5 μm and 10 μm core diameter capillaries.

First, we investigate unspliced capillary fibers where no single-mode fiber is spliced up front. This enables the most flexible mode-matching of the incoming focused laser beam to the fundamental fiber mode in the capillary due to free space coupling. It is very important for the generation of broad supercontinua that mostly the fundamental mode is excited due to the smallest effective mode area which results in the highest nonlinearity.

Our second approach is an integrated liquid-filled capillary with a standard single-mode fiber spliced in front of the capillary. A key requirement for the splicing is the maintenance of single-mode operation despite the multi-mode capability of the capillaries. This is achieved by our newly developed mechanical splicing method. The mode profile at the fiber end is detected with an InGaAs camera as illustrated in Fig. 2 for an unspliced as well as for a spliced capillary fiber with 10 μm core diameter for a pump wavelength of 1510 nm to demonstrate single-mode operation. Further benefits of the integrated device are the avoidance of evaporation of the core liquid and the robustness and stability of the splice. Moreover, it is easier to couple into a fused silica fiber compared to a liquid-filled capillary.

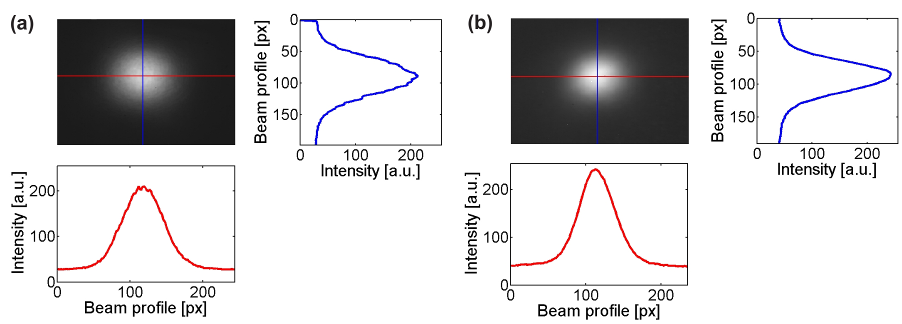

Fig. 2. Mode field distribution for a CS_2-filled capillary fiber with a core diameter of 10 μm for a pump wavelength of 1510 nm recorded with a InGaAs camera. Single-mode operation was achieved for (a) unspliced as well as for (b) spliced capillary fibers.

3. Experimental setup

The experimental setup is shown in Fig. 3. A home built Yb:KGW femtosecond laser oscillator [15] at a wavelength of 1030 nm and a fiber-feedback optical parametric oscillator (OPO) at wavelengths of 1510 nm and 1685 nm are used as pump sources for the supercontinuum generation. The Yb:KGW oscillator emits pulses with a duration of 450 fs at full width at half

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maximum at 41 MHz repetition rate. The OPO is pumped by the Yb:KGW oscillator and delivers tunable pulses between 1380 nm and 2030 nm with a duration of 350 fs at repetition rate of 41 MHz. The pulse shapes of the Yb:KGW oscillator and the OPO are hyperbolic secant as determined by an autocorrelation measurement, whereas the OPO pulse is slightly negatively chirped.

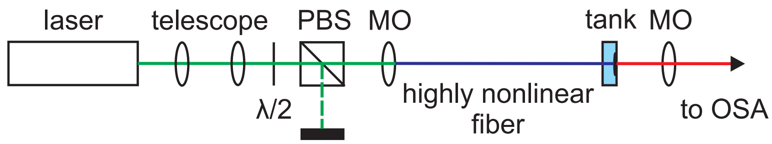

Fig. 3. Schematic diagram of the experimental setup for supercontinuum generation. PBS: polarizing beam splitter; MO: microscope objective; OSA: optical spectrum analyzer.

A telescope is used to adapt the beam waist to the different core diameters of the highly nonlinear fibers (either unspliced or integrated capillaries). With a half-wave plate in combination with a polarizing beam splitter (PBS), the power launched into the fiber can be adjusted. Depending on the fiber core diameter different aspherical lens objectives (16x and 20x) are utilized to couple and optimize the beam waist to the fundamental fiber mode. The liquid filling of the fiber capillaries happens by capillary force from a liquid-filled tank and no special equipment is needed. The fiber end is placed closely to the glass plate window of the liquid-filled tank to effectively collimate the output beam with a 40x microscope objective. The average output power is measured behind the outcoupling objective. Finally, the laser beam is focused directly into an optical spectrum analyzer (Ando AQ-6315E or Yokogawa AQ6375) with a measuring range from 350 nm up to 1750 nm and 1200 nm up to 2400 nm, respectively.

The best supercontinuum results have been achieved for fiber lengths of our liquid-filled capillaries between 15 cm and 20 cm. A further increase in length does not cause significantly broader spectra.

4. Measurement results for unspliced fiber capillaries

Figure 4 shows the measured supercontinuum spectra and corresponding pump laser spectra at the three investigated wavelengths of 1030 nm, 1510 nm, and 1685 nm for the unspliced fiber capillaries. Due to the direct free space coupling we can selectively excite the fundamental mode which results in broad white light spectra for the 5 μm (green) and 10 μm (red) capillaries. The overall achieved transmission efficiencies through the liquid-filled capillary fibers, measured before the incoupling and behind the liquid-filled tank are about $75-80\%$ at 1030 nm, $60-75\%$ at 1510 nm, and $40-60\%$ at 1685 nm for the 5 μm and 10 μm core diameters. The efficiencies are quite good despite the fact that the profile of the CS_2-air interface at the fiber end into which light is coupled in is slightly bent in case of the 10 μm core diameter capillary [see Fig. 5]. The decrease in transmission comes mainly from the mode profile of the OPO which is worse at longer wavelengths. The spectral broadening increases for higher pump wavelengths especially for the 5 μm capillary due to the better dispersion properties in the higher wavelength range.

In the case of the 1030 nm pump wavelength both fibers are clearly in the normal dispersion region [see Fig. 1(b)] which leads to relatively fast pulse broadening in the time domain and to relatively small spectral broadening. The main mechanism responsible for the large third order nonlinearity of CS_2 is a non-instantaneous molecular response mainly attributed to molecular reorientation [16, 17]. It originates from the tendency of anisotropic molecules to become aligned in the electric field of an applied optical wave. The strength of the delayed molecular response depends on the pulse duration and its relation to the relaxation time of the liquid,

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which lies for CS_2 in the ps-range [17]. The fraction of instantaneous electronic contribution to the nonlinear refractive index for our laser parameters is around 15 % [9]. The contribution

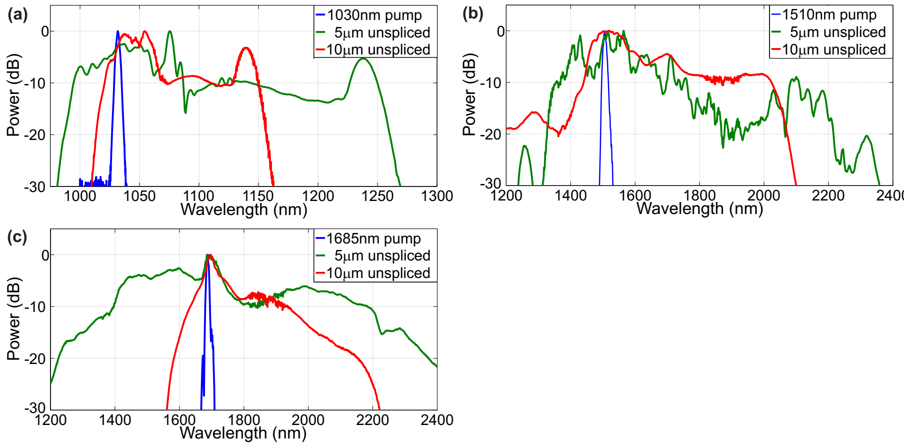

Fig. 4. Normalized supercontinuum spectra for unspliced capillary fibers of 5 μm (green), and 10 μm (red) core diameters filled with CS_2 and corresponding pump laser spectra (blue) at a center wavelength of (a) 1030 nm, (b) 1510 nm, and (c) 1685 nm. The achieved average output powers were (a) 35 mW, (b) 24 mW, (c) 17 mW and (a) 70 mW, (b) 90 mW, (c) 25 mW for the 5 μm and 10 μm capillary fibers, respectively.

of intramolecular Raman effect can be neglected for our pulse duration of $> 350\,fs$ [3, 18, 19]. The nonlinear parameter $\gamma = 2\pi n_{2I}/(\lambda_0 A_{\text{eff}})$ with the pump wavelength λ_0 and the effective mode area A_{eff} is on the order of $2\,(\text{Wm})^{-1}$ for our capillary fibers. The spectral broadening in the liquid-filled fiber capillaries due to the effect of self-phase modulation is accompanied by the effect of the slow material response of CS_2 [19, 20]. The delayed nature of the nonlinear response leads to an energy transfer to higher wavelengths which is accompanied by a large red-shift of the pulse spectrum [19]. This can be observed in the spectra of Fig. 4. However, due to the extremely large nonlinear refractive index of CS_2 [9], only low average powers, e.g., lower than 50 mW, are necessary to realize a broadening of more than 250 nm in the case of the 5 μm capillary.

The achieved output average powers of 35 mW and 70 mW for the 5 μm and 10 μm core diameter capillaries at a wavelength of 1030 nm correspond to peak powers of 1.7 kW and 3.4 kW, respectively. The limitation of the maximum peak power arise from a sudden drop in

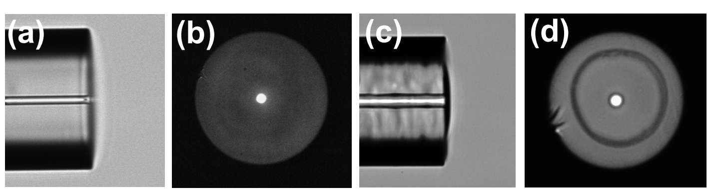

Fig. 5. Microscope images of the fiber end into which light is coupled in the unspliced capillaries. Side view of (a) 5 μm and (c) 10 μm core diameter capillaries, respectively, and the corresponding top views (b), (d). For the 10 μm capillary the fiber end face is wetted by CS_2 and the profile of the CS_2-air interface is slightly bent which do not diminish the coupling efficiencies. For the 5 μm capillary the end face is not wetted and CS_2 is only located in the core.

transmission due to the formation of air bubbles in the liquid core at the capillary entrance which we monitored with a microscope. This reproducible behavior was also observed in the study of Kieu et al. [6]. They assumed multi-photon absorption to create thermal effects which increase the temperature above the boiling temperature of CS_2 (46 °C [21]). A further possible reason could be dissolved gases in the liquid which also cause bubble formation at a solid-liquid interface achieved by heat.

Pumping at higher wavelengths should overcome the issue with multi-photon absorption and hence higher peak powers should be possible. For the 5 μm core diameter capillary the achievable output peak power remains nearly the same as for the shorter pump wavelength whereas the spectral broadening could be enhanced dramatically. The spectrum spans from 1300 nm up to 2200 nm [see Fig. 4(b)]. A further increase in the pump wavelength to 1685 nm increases the broadening for the 5 μm capillary to a bandwidth of 1200 nm, ranging from 1200 nm to 2400 nm as shown in Fig. 4(c). This arises from the fact that the pump wavelength comes close to the zero-dispersion wavelength. Hence, wavelength components are shifted into the anomalous dispersion region where soliton dynamics [1] in combination with a very strong self-phase modulation component which is mainly induced by the delayed molecular effect [3, 19] are responsible for the large spectral broadening. Unfortunately, the decreasing beam profile quality of the OPO for higher output power hinders a further increase in the achievable peak power in the 5 μm capillary which is 1.1 kW.

In the case of the 10 μm core diameter capillary we could increase the maximum average output power to 90 mW (corresponding to 5.5 kW peak power) for the longer pump wavelength of 1510 nm. The spectral broadening of the 10 μm capillary is largest at the pump wavelength of 1510 nm, stretching from below 1200 nm (limit of the Yokogawa spectrometer) up to 2100 nm. Here a further increase in the pump wavelength can not increase the broadening because the wavelength components are still in the normal dispersion region.

5. Fabrication and measurement results for integrated fiber capillaries

For an improved handling of the liquid-filled fiber capillaries that maintained single-mode op-

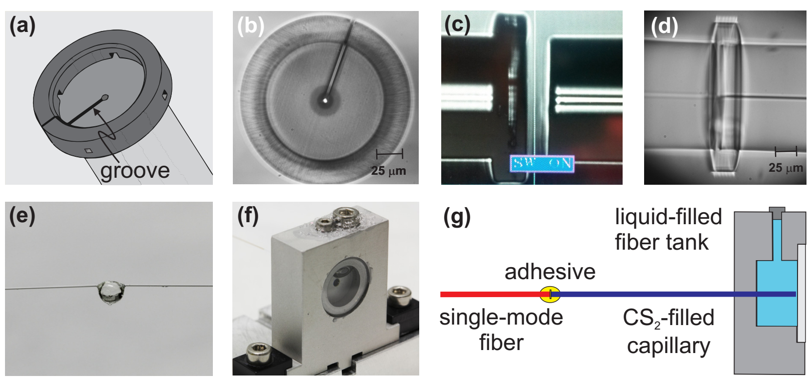

Fig. 6. Schematic of the fabrication process for an integrated liquid-filled device. (a) Design of the ferrule on a standard single-mode fiber consisting of a base plate (light gray) and a ring (dark gray). (b) Top view of the fabricated ferrule by direct laser writing. (c) Assembly of the single-mode fiber with ferrule (left) and the CS_2-filled capillary (right) via a camera. (d) Mechanically spliced fibers aligned by the fabricated ferrule on the single-mode fiber. (e) Strengthening of the splice with UV-adhesive. (f) Liquid-filled tank on the opposite site of the splice. The capillary is put into the tank before the splice takes place. (g) Layout of the whole highly nonlinear integrated liquid-filled device.

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eration we developed a new mechanical splicing method based on an alignment sleeve that is written on a single-mode fiber by 3D femtosecond direct laser writing using a dip-in photoresist and the system "Nanoscribe" from Nanoscribe [see Fig. 6] [22]. The mechanism of direct laser writing is based on two photon absorption of a photo-sensitive material in a tightly focused spot of a fs laser. As a printing medium we use a negative-tone photoresist. The laser spot is scanned over the sample, writing the desired object by exposing the polymer layer by layer. The polymerization is triggered only in the focal point volume. Hence, very accurate 3D microstructures can be fabricated. The writing time for our ferrule takes about 12 minutes using a galvo scanner. Afterwards, the sleeved fiber is put into a solvent for 15 min to remove remaining unpolymerized photoresist.

The ferrule written on the SMF consists of a 10 μm thick base plate and a ring with an outer diameter of 175 μm and an inner diameter of 125 μm, adapted to the capillary cladding diameter [Fig. 6(a)]. The base plate is directly fabricated on the SMF and therefore ensures proper connection. The base plate features a central hole that can be adjusted to the hole diameter of the capillary [Fig. 6(b)]. The height of the ferrule is 35 μm. Four side holes and a central groove enable that remaining air can flow out when connecting the sleeved SMF to the liquid-filled capillary fiber. The adjustment can be monitored with a camera [Fig. 6(c)]. The central hole in the base plate is also filled with the liquid as soon as both fibers touch each other, forming a direct liquid fused-silica interface [Fig. 6(d)]. To protect and strengthen the splice, a drop of UV-glue is put onto the splice [Fig. 6(e)]. The other fiber end remains in a sealed liquid-filled tank and hinders the evaporation of the liquid CS_2 [Fig. 6(f)]. The schematic scheme of the whole highly nonlinear integrated liquid-filled fiber device can be seen in Fig. 6(g). The device is stable for weeks and also the splice is very robust which ensures a flexibility in transportation and handling. Due to the easy variation of the ferrule diameter the method can also be used to splice other fiber types.

In Fig. 7 measurement results for the spliced fiber capillaries at the three investigated pump wavelengths of 1030 nm, 1510 nm, and 1685 nm are illustrated together with the pump laser spectra. As in the case of the unspliced fiber the 5 μm core diameter capillary results in the stronger broadening compared to the 10 μm capillary. At the highest pump wavelength of

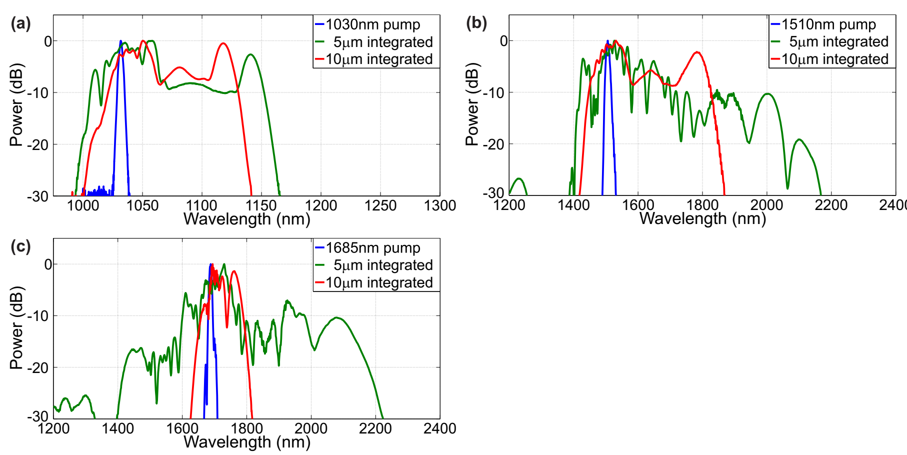

Fig. 7. Normalized supercontinuum spectra for integrated capillary fibers of 5 μm (green), and 10 μm (red) core diameters filled with CS_2 and corresponding pump laser spectra (blue) at a center wavelength of (a) 1030 nm, (b) 1510 nm, and (c) 1685 nm. The achieved average output powers were (a) 14 mW, (b) 25 mW, (c) 14 mW and (a) 75 mW, (b) 43 mW, (c) 20 mW for the 5 μm and 10 μm capillary fibers, respectively.

1685 nm we obtain the largest spectral broadening, stretching from 1400 nm up to 2200 nm. However, the achieved white-light spectra with the spliced capillaries are not as broad as for the unspliced capillaries. This arises from the mode mismatch between the SMF and the liquid-filled capillary which leads to a small extent also to the excitation of higher order modes, which diminish the nonlinearity resulting in a reduction of spectral broadening. We optimize the mode matching by using single-mode fibers with different core size, e.g., UHNA3, HI1060, and SMF28. The best results were achieved with Corning SMF HI1060 for both capillary core diameters.

The transmission efficiencies through the integrated fiber capillaries are $65-72\,\%$ at 1030 nm, $50-72\,\%$ at 1510 nm, and $35-45\,\%$ at 1685 nm for the 5 μm and 10 μm core diameters. The better handling of the integrated fiber device is at the cost of somewhat lower spectral broadening, whereas the broadening behavior is similar to the unspliced capillaries. The impact of the beneficial dispersion property of the 5 μm core diameter capillary still results in a 800 nm broad supercontinuum at a pump wavelength of 1685 nm. The achieved average output power in this case is 14 mW (corresponds to 0.9 kW peak power) which is comparable to the unspliced case. The maximum output power could not be increased further despite the avoidance of direct focusing into the liquid, and bubble formation remains a limitation. Placing the integrated fiber onto a cooled copper block ($T = 12\,°\mathrm{C}$) can slightly increase the peak power.

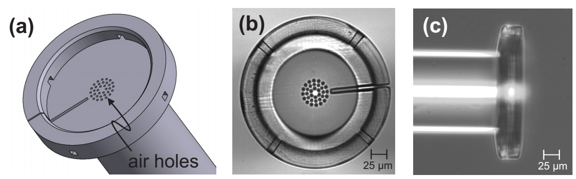

Fig. 8. (a) Schematic of the ferrule design for a mechanical splice between two heat sensitive solid-core fibers (e.g. ZBLAN-chalcogenide). The design is similar to the integrated fiber capillaries composed of a base plate with some air holes to create a step-index profile and an outer ring. (b) Top view and (c) side view of the fabricated ferrule by direct laser writing onto a ZBLAN-fiber. The inner diameter of the outer ring has been adjusted to a chalcogenide fiber with a cladding diameter of 175 μm.

6. Conclusion

We have demonstrated supercontinuum generation in the mid-IR wavelength region in 5 μm and 10 μm core diameter capillary fibers filled with the highly nonlinear liquid CS_2. The large spectral broadening ranges from 1200 nm up to 2400 nm due to beneficial dispersion properties of the investigated fibers. Despite the multi-mode capability at our three different pump wavelengths of 1030 nm, 1510 nm, and 1685 nm we were able to selectively excite mostly the fundamental mode in unspliced as well as integrated capillaries. Therefore, we used a novel mechanical splicing method based on 3D femtosecond direct laser writing. The method is rather flexible and can be extended to heat sensitive fibers with diameters other than the 125 μm cladding standard [see Fig. 8]. The current limitation in the achievable output peak power caused by the generation of cavitation bubbles may be suppressed by degasification of the liquid or by using pressurized liquid chambers as in the work of Fanjoux et al. [8].

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