

T. Ritari, J. Tuominen, H. Ludvigsen, J. C. Petersen, T. Sørensen, T. P. Hansen, and H. R. Simonsen. 2004. Gas sensing using air-guiding photonic bandgap fibers. *Optics Express*, volume 12, number 17, pages 4080-4087.

© 2004 Optical Society of America (OSA)

Reprinted with permission.

Gas sensing using air-guiding photonic bandgap fibers

T. Ritari, J. Tuominen, and H. Ludvigsen

*Fiber-Optics Group, Department of Electrical and Communications Engineering, Helsinki University of Technology,
P.O.Box 3500, FI-02015 HUT, Finland
Tuomo.Ritari@hut.fi*

J. C. Petersen

Danish Institute of Fundamental Metrology, Matematiktorvet Building 307, DK-2800 Kgs. Lyngby, Denmark

T. Sørensen

Research Center COM, Technical University of Denmark, DTU Building 345W, DK-2800 Kgs. Lyngby, Denmark

T. P. Hansen* and H. R. Simonsen

*Crystal Fibre A/S, Blokken 84, DK-3460 Birkerød, Denmark
) Also with Research Center COM, Technical University of Denmark

Abstract: We demonstrate the high sensitivity of gas sensing using a novel air-guiding photonic bandgap fiber. The bandgap fiber is spliced to a standard single-mode fiber at the input end for easy coupling and filled with gas through the other end placed in a vacuum chamber. The technique is applied to characterize absorption lines of acetylene and hydrogen cyanide employing a tunable laser as light source. Measurements with a LED are also performed for comparison. Detection of weakly absorbing gases such as methane and ammonia is explored.

©2004 Optical Society of America

OCIS codes: (060.2370) Fiber optics sensors, (230.3670) Light-emitting diodes, (230.3990) Microstructure devices, (300.1030) Absorption

References and links

1. S. Sudo, I. Yokohama, H. Yasaka, Y. Sakai, and T. Ikegami, "Optical fiber with sharp optical absorption by vibrational-rotational absorption of C₂H₂ molecules," *IEEE Photonics Technol. Lett.* **2**, 128-131 (1990).
2. W. Jin, G. Stewart, and B. Culshaw, "Prospects for fibre-optic evanescent-field gas sensors using absorption in the near-infrared," *Sens. and Act. B* **38-39**, 42-47 (1997).
3. J. Harrington, "A review of IR transmitting hollow waveguides," *Fiber Integr. Opt.* **19**, 211-227 (2000).
4. T. M. Monro, D. J. Richardson, and P. J. Bennett, "Developing holey fibres for evanescent field devices," *Electron. Lett.* **35**, 1188-1189 (1999).
5. T. M. Monro, W. Belardi, K. Furusawa, J. C. Baggett, N. G. R. Broderick, and D. J. Richardson, "Sensing with microstructured optical fibres," *Meas. Sci. Technol.* **12**, 854-858 (2001).
6. Y. L. Hoo, W. Jin, H. L. Ho, D. N. Wang, and R. S. Windeler, "Evanescent-wave gas sensing using microstructure fiber," *Opt. Eng.* **41**, 8-9 (2002).
7. Y. L. Hoo, W. Jin, C. Shi, H. L. Ho, D. N. Wang, and S. C. Ruan, "Design and modeling of a photonic crystal fiber gas sensor," *Appl. Opt.* **42**, 3509-3515 (2003).
8. G. Pickrell, W. Peng, and A. Wang, "Random-hole optical fiber evanescent-wave gas sensing," *Opt. Lett.* **29**, 1476-1478 (2004).
9. R. F. Cregan, B. J. Mangan, J. C. Knight, T. A. Birks, P. St. J. Russell, P. J. Roberts, and D. C. Allan, "Single-mode photonic band gap guidance of light in air," *Science* **285**, 1537-1539 (1999).
10. J. Lægsgaard, N. A. Mortensen, J. Riishede, and A. Bjarklev, "Material effects in air-guiding photonic bandgap fibers," *J. Opt. Soc. Am. B.* **20**, 2046-2051 (2003).
11. G. Humbert, J. C. Knight, G. Bouwmans, P. S. J. Russell, D. P. Williams, P. J. Roberts, and B. J. Mangan, "Hollow core photonic crystal fibers for beam delivery," *Opt. Express* **12**, 1477-1484 (2004).
12. B. J. Mangan, L. Farr, A. Langford, P. J. Roberts, D. P. Williams, F. Couny, M. Lawman, M. Mason, S. Coupland, R. Flea, H. Sabert, T. A. Birks, J. C. Knight, and P. St. J. Russell, "Low loss (1.7 dB/km) hollow

- core photonic bandgap fiber,” in *Proceedings of Optical Fiber Communication Conference*, postdeadline paper PDP24, Los Angeles, USA, (2004).
13. T. P. Hansen, J. Broeng, C. Jakobsen, G. Vienne, H. R. Simonsen, M. D. Nielsen, P. M. W. Skovgaard, J. R. Folkenberg, and A. Bjarklev, “Air-guiding photonic bandgap fibers: Spectral properties, macrobending loss and practical handling,” *J. Lightwave Technol.* **22**, 11-15 (2004).
 14. T. Ritari, J. Tuominen, J.C. Petersen, T.P. Hansen, and H. Ludvigsen, “Miniature wavelength references based on gas-filled photonic bandgap fibers”, in *Proceedings of European Conference on Optical Communication*, paper Mo3.3.1, Stockholm, Sweden (2004).
 15. T. Dennis, E. A. Curtis, C. W. Oates, L. Hollberg, and S. L. Gilbert, “Wavelength references for 1300-nm wavelength-division multiplexing,” *J. Lightwave Technol.* **20**, 804-810 (2002).
 16. A. Schmohl, A. Miklós, and P. Hess, “Detection of ammonia by photoacoustic spectroscopy with semiconductor lasers,” *Appl. Opt.* **41**, 1815-1823 (2002).
 17. T. J. Stephens, R. R. J. Maier, J. S. Barton, and J. D. C. Jones, “Fused silica hollow-core photonic crystal fiber for mid-infrared transmission,” in *Proceedings of Conference on Lasers and Electro Optics*, postdeadline paper CPDD4, San Francisco, USA (2004).
 18. T. M. Monro, Y. D. West, D. W. Hewak, N. G. R. Broderick, and D. J. Richardson, “Chalcogenide holey fibres,” *Electron. Lett.* **36**, 1998–2000 (2000).
-

1. Introduction

Optical fibers used for gas sensing offer clear advantages such as immunity to electromagnetic interference, small size, low cost and the possibility for distributed measurements. Different fiber designs including fibers with a small hole in the center of the core [1] and D-shaped optical fibers [2] have previously been employed in gas sensing. However, such fiber sensors suffer from a poor overlap between the gas volume and the mode field of the propagating light, which results in weak absorption and therefore long length of fibers are required. Hollow optical waveguides have also been used but they are usually multi-mode and their losses are high, which limits the practical waveguide length to a few meters [3].

The overlap between the gas and mode field can be improved by using a photonic bandgap fiber (PBF) [4-8]. In this type of fiber, the light is confined within the air core by a two-dimensional photonic bandgap formed by the periodic structure of the cladding allowing transmission over a limited wavelength range [9]. It has been shown that in PBFs more than 98 % of the guided mode field energy can propagate in the air regions of the fiber [10-12]. Therefore, by filling the air holes of a PBF with gas a significant increase in the overlap between the volume of the gas and the mode field of the light propagating along the fiber can be obtained thereby reducing considerably the length of fiber needed. Furthermore, the relatively large core of a PBF can be filled in a short time, thus improving the response time of the sensor apparatus. Besides, PBFs are highly insensitive to bending, which allows for the construction of compact devices [13]. PBF based fiber sensors could play a major role in the field of spectroscopy (e.g., gas detection) and sealed gas-filled PBFs in telecommunication (wavelength reference) [14].

In this paper, we investigate the feasibility of using PBFs in gas detection and demonstrate the high sensitivity of the technique with acetylene. The absorption spectra are measured using both a tunable laser and a light emitting diode (LED) in the 1500 nm region. The results are compared with measurements performed using a conventional gas absorption cell. Furthermore, the technique is also applied in the 1300 nm region using a different PBF to characterize methane which has weak absorption lines difficult to detect using conventional methods.

2. Experimental setup

The PBFs used in the experiments exhibit a triangular cladding-hole structure shown in the microscope images of Fig. 1. In the following, the fibers are labeled as PBF1300 and PBF1500 according to their transmission windows. In both fibers, the core was formed by removing seven silica tubes from the center of the fiber preform. The characteristic dimensions of the fibers are summarized in Table 1.

Table 1. Characteristics of the PBFs.

	PBF1300	PBF1500
Core size (μm)	11.6	10
Pitch (μm)	3.1	3

The normalized transmissions for a 2-m long sample of PBF1300 and 3 m of PBF1500 as a function of wavelength are presented in Fig. 2. The wavelength ranges of photonic bandgap guidance are approximately 1240-1460 nm and 1400-1600 nm for PBF1300 and PBF1500, respectively. To improve the coupling efficiency to the bandgap fibers, the input end of the PBFs was spliced to a standard single-mode fiber (SMF) terminated by a fiber connector. The splicing losses were estimated to be ~ 1 dB and are mainly caused by mode field mismatch and light reflection from the silica-air interface. The loss of PBF1300 was estimated to be less than 0.1 dB/m within the wavelength range of 1250-1380 nm and the loss of PBF1500 less than 0.2 dB/m within the wavelength range of 1460-1575 nm.

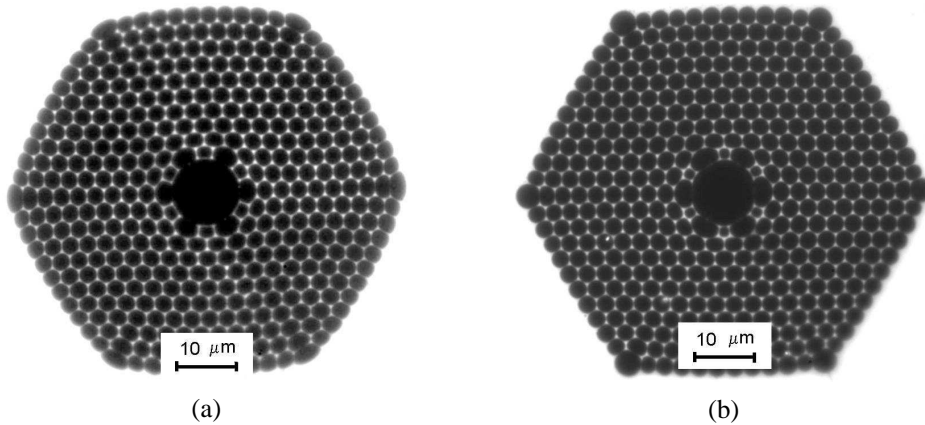


Fig. 1. Microscope images of (a) PBF1300 and (b) PBF1500.

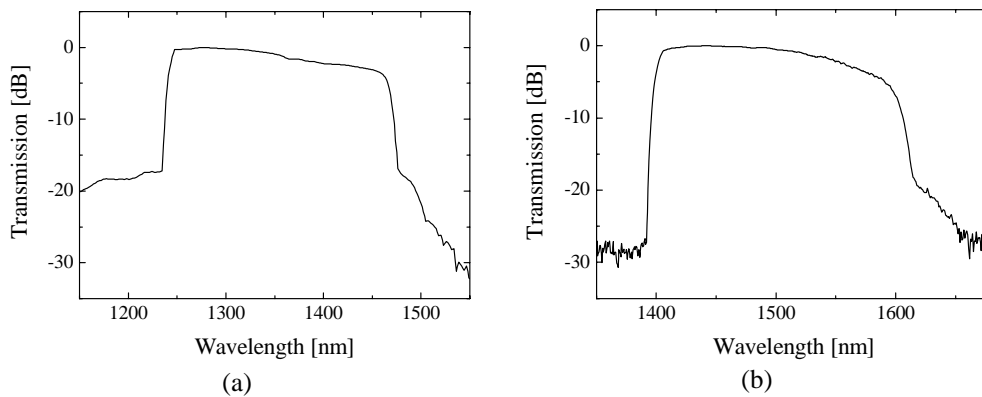


Fig. 2. Spectral transmission of (a) a 2 m long PBF1300 and (b) a 3 m long PBF1500.

The experimental setup used to fill the PBFs with gas and to perform absorption measurements is depicted in Fig. 3. The purities of all the gases investigated were specified by the manufacturers to be $\geq 99\%$. The open end of the PBF is butt-coupled to a multi-mode fiber (MMF) using a V-groove placed inside a vacuum chamber. A rotary pump with a

pumping rate of 45 l/s was employed to evacuate the chamber and a conventional vacuum system was utilized for obtaining the desired pressure in the fibers. The gap between the butt-coupled fibers was adjusted to $\sim 50 \mu\text{m}$ for efficient filling while keeping good output coupling efficiency. The gas absorption was measured by coupling light from a tunable laser or LED into the SMF spliced to the PBF and recording the transmission at the output of the MMF as a function of wavelength. In the case of a tunable laser, the light at the output of the MMF was detected with a germanium photodetector and ten percent of the laser output power was used to monitor the wavelength accurately. The data were collected with a computer. The experiments were conducted in a temperature-stabilized environment at $22 \pm 1 \text{ }^\circ\text{C}$. When performing measurements with the LED as the light source, the absorption spectra were recorded using a calibrated optical spectrum analyzer (OSA).

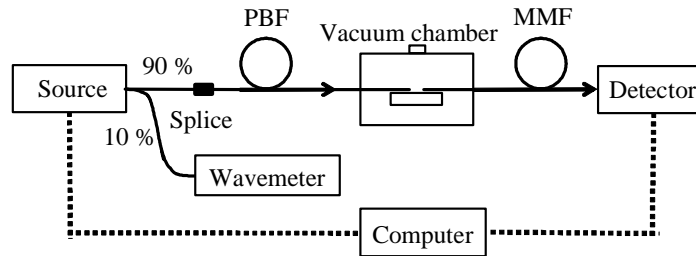


Fig. 3. Experimental setup for filling PBFs with gas and absorption measurements.

3. Fiber filling and evacuation dynamics

We first investigated the dynamics of the filling and evacuation processes which are of prime importance for practical sensor applications [7]. Acetylene is known to have a strong absorption band in the 1550 nm region and is therefore particularly suited for exploring the capabilities of PBFs for sensing purposes. The filling process was investigated by monitoring the light transmitted through 0.8 m of PBF1500 for the $^{12}\text{C}_2\text{H}_2$ lines at 1531.588 and 1521.060 nm as a function of time. The experiment was performed at two different pressures for the two lines: 10 and 113 mbar, respectively. The normalized transmission as a function of time for the two absorption lines is displayed in Figs. 4 and 5. Exponential fits plotted as solid lines are also shown. The time constants of the filling processes obtained from the fits are approximately equal to 6 s and 4 s, respectively.

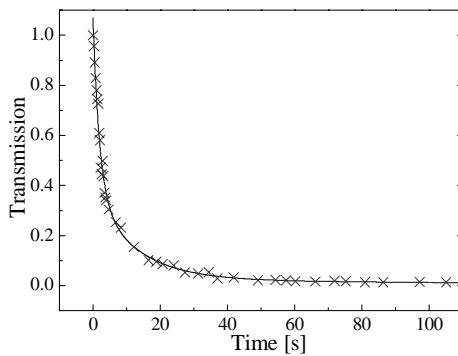


Fig. 4. Normalized transmission of $^{12}\text{C}_2\text{H}_2$ line at 1531.588 nm as a function of time in PBF1500 recorded using a tunable laser while filling the fiber with gas to 10 mbar.

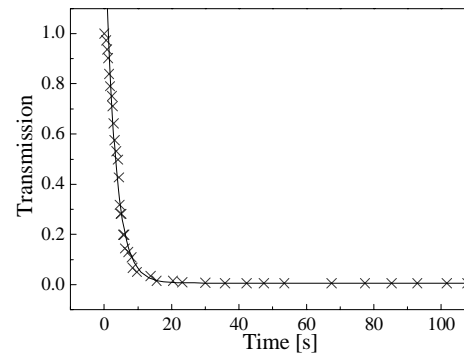


Fig. 5. Normalized transmission of $^{12}\text{C}_2\text{H}_2$ line at 1521.060 nm as a function of time in PBF1500 recorded using a tunable laser while filling the fiber with gas to 113 mbar.

The evacuation times of the fibers for the pressures of 10 mbar and 113 mbar were also investigated using the aforementioned rotary pump and found to be approximately 30 min and 1 hour, respectively. We can conclude that the use of higher pressure results in a shorter filling time and a longer evacuation time. An experiment was performed where a fiber filled with acetylene at 113 mbar was opened to ambient air. Similar to the experiments above an absorption line was monitored using a tunable laser. It was observed that approximately 14 hours elapsed before the absorption line could no longer be observed. Furthermore, we observed that the gas filling and evacuation processes are also strongly influenced by the molecular species, the length of the fiber and the pump speed. A more detailed study of filling and evacuation times is in progress.

4. Absorption measurements of acetylene

A 1-m long piece of PBF1500 was filled with acetylene utilizing the experimental setup described in section 2. The absorption spectrum was first measured using the tunable laser with a wavelength step of 1 pm. Figure 6 shows the spectrum of the P-branch of the $\nu_1+\nu_3$ band of $^{12}\text{C}_2\text{H}_2$ measured at a pressure of 10 mbar. The signal-to-noise ratio of the measurement exceeds 20 dB. The strong absorption lines show the potential of PBFs for high sensitivity gas detection.

To compare the absorption measurements in PBFs with the more traditional methods, we measured the P-branch absorption of $^{12}\text{C}_2\text{H}_2$ in a 1 m long cell using the same tunable laser. The results are presented in Fig. 7. The signal-to-noise ratio is better in the case of the standard open cell which could be expected since PBFs are sensitive to temperature changes, vibrations and reflections from the fiber ends. The temperature and pressure in the cell and PBF were close to identical yielding roughly the same molecular density. Since the same laser power was employed in the two measurements and this power is far from the saturation power, similar strengths for the absorption lines are expected in the two experiments. However, due to coupling losses into the fiber and none perfect polarization of the beam passing the Brewster windows of the open cell the absolute power in the absorption cells is not suited for an absolute comparison. A closer inspection of the background noise in the fiber measurement shows an interference pattern that we believe to be due to the polarization properties and the aligning of the PBF.

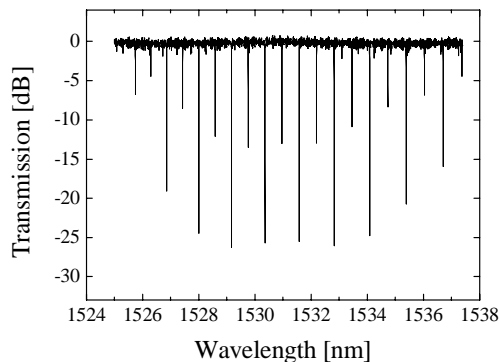


Fig. 6. Normalized absorption spectrum of the P-branch of $^{12}\text{C}_2\text{H}_2$ at 10 mbar in a 1 m long PBF1500 measured using a tunable laser (step size 1 pm).

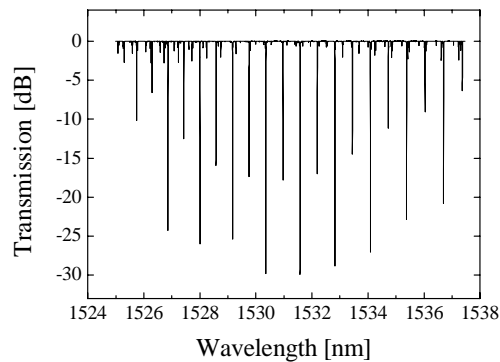


Fig. 7. Normalized absorption spectrum of the P-branch of $^{12}\text{C}_2\text{H}_2$ at 10 mbar in a 1 m long absorption cell measured using a tunable laser (step size 1 pm).

Moderate wavelength accuracy is usually sufficient to identify molecular species so that a LED can be utilized as a light source instead of a tunable laser to measure the absorption spectra. We measured the absorption spectrum of the R-branch of the $\nu_1+\nu_3$ band of $^{12}\text{C}_2\text{H}_2$

using a LED. The spectrum was recorded with an OSA for a pressure of 200 mbar in a 1 m long PBF1500 (see Fig. 8). For comparison, the same absorption spectrum of $^{12}\text{C}_2\text{H}_2$ at a pressure of 10 mbar measured with the tunable laser is displayed in Fig 9. The lower resolution of the OSA results in weaker absorption lines and therefore the measurements with the LED were conducted at higher pressure. The spectrum shown in Fig. 8 has been normalized to the emission spectrum of the LED. The small variations in the strengths of the absorption lines are mainly caused by the variation in the transmitted LED power between the background and the absorption spectra.

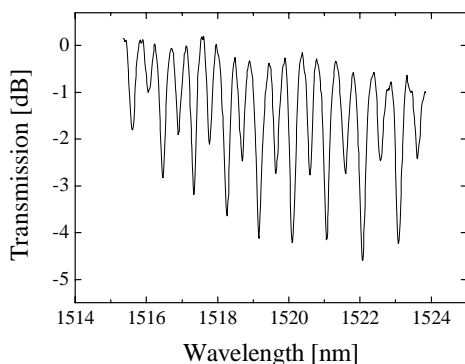


Fig. 8. Normalized absorption spectra of R-branch of $^{12}\text{C}_2\text{H}_2$ in a 1 m long PBF1500 measured using a LED. The resolution of the OSA is 0.1 nm. The lines appear broader due to the limited resolution of the OSA.

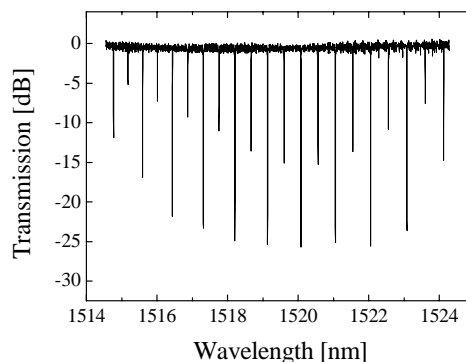


Fig. 9. For comparison, the same spectrum recorded using a laser (step size 1 pm) and a reduced pressure of 10 mbar.

5. Absorption measurements of hazardous gases

Employing the same setup as for acetylene, we subsequently investigated the possibility of monitoring gases such as hydrogen cyanide (HCN), methane (CH_4) and ammonia (NH_3) [15,16]. Emission of such gases is indeed important to detect for health, safety and environmental reasons. Due to a weak absorption of methane and ammonia (approximately two orders of magnitude weaker than acetylene), these gases are difficult to detect using other methods [1,2]. Whereas, it is expected that the utilization of air-guiding photonic crystal fibers for the detection of weakly absorbing molecules would highly increase the sensitivity of the sensor due to the significant improvement in overlap between the guided mode and the gas.

We first filled a 1-m long PBF1500 with hydrogen cyanide (H^{13}CN isotope) at a pressure of ~ 50 mbar. Figure 10 illustrates the R-branch of the $2\nu_3$ band of H^{13}CN consisting of more than 25 strong vibration-rotation absorption lines in the 1525-1545 nm region measured using a LED and an OSA. In this wavelength region, the spectral difference between this molecule and the main isotope H^{12}CN is a shift of the absorption lines by approximately 9 nm towards lower wavelengths.

The detection of methane was also investigated by employing a 10 m long piece of PBF1300 and a pressure of 630 mbar. Figure 11 shows part of the R-branch of the $\nu_2+2\nu_3$ combination band measured using a LED and an OSA. Note that due to the weaker absorption of methane compared to hydrogen cyanide, a higher pressure and a longer fiber length were used. The spectrum is known to consist of multiplets, which are not completely resolved in our recording. The strongest feature is the Q-branch, near 1330 nm. This branch can be partly resolved by using a laser source with a step size of 0.1 pm. However, for monitoring purposes the Q-branch feature is unique and the use of a LED will generally be sufficient.

A partial spectrum of ammonia has also been recorded at a pressure of 32 mbar near 1500 nm using the 1 m long PBF1500 and a laser source. Ammonia has a very congested spectrum

in the 1500 nm region and a LED based spectrum may not be sufficient for proper identification. Further studies of ammonia are in progress and in particular filling dynamics is of interest due to the high adhesion of ammonia on glass walls.

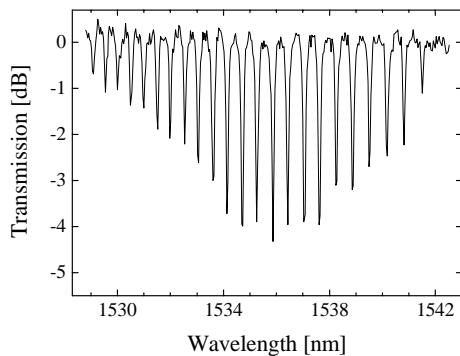


Fig. 10. Normalized absorption spectrum of H^{13}CN in a 1 m long PBF1500 recorded using a LED. The resolution of the OSA is 0.1 nm.

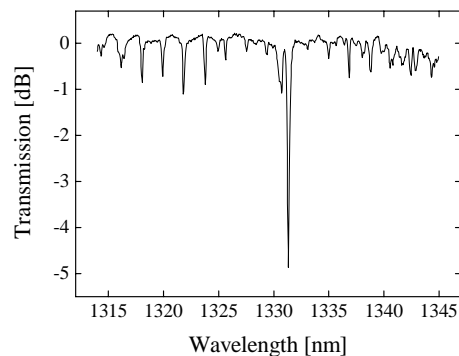


Fig. 11. Normalized absorption spectrum of CH_4 in a 10 m long PBF1300 recorded using a LED. The resolution of the OSA is 0.1 nm.

6. Discussion

In practical applications, parameters such as sensitivity and fiber length need to be considered in detail in order to optimize a PBF sensor. The relationship between absorption length (fiber length), gas concentration and light intensity is given by the Beer-Lambert law [7]. In addition, in order to minimize the response time of the sensor, the fiber should be as short as possible while still long enough to provide a sufficient signal. The optimum length depends on the molecular species to be monitored and the amount of gas present in the environment. For gases with weak absorption lines or in low concentration, an increased sensitivity can be obtained by using longer fiber length. However, the attenuation increases with the length of the fiber.

Effects limiting the sensitivity of the detection are mainly fiber loss and background noise which is expected to result from the polarization properties and the aligning of the PBF. Furthermore, care has to be taken in the evacuation of the fiber since the remaining gas in the fiber may contaminate the subsequent measurements. In field use, dirt and water blocking the holes are also issues to be solved. In practice, reflection measurements could be preferable since launch and detection will occur at the same position. Moreover, PBFs are currently available in the wavelength range from 400 to 2550 nm with varying losses [17]. This broad wavelength region gives the potential for monitoring a large range of gases. The gas detection range could be further extended by using special compound glasses that are transparent in the mid infrared region [18].

7. Conclusion

We have demonstrated that air-guiding PBFs can be used in sensing both strongly (acetylene/hydrogen cyanide) and weakly (methane/ammonia) absorbing gases. The advantages of using PBFs as gas sensors include large overlap and long optical path interaction between the gas and light mode field and require only a small sample volume. Furthermore, PBFs are insensitive to bending, may be connected to standard fiber-optic instruments using advanced splicing techniques [13] and the design of a PBF-based sensor is simple compared to multi-pass gas cells that can be cumbersome to operate. PBFs may find applications in analyzing gas samples using small gas volumes and in remote safety monitoring of reactive or poisonous gases.

Acknowledgments

The work has been financially supported by the Academy of Finland, the Nordic Academy for Advanced Study, and the graduate school of Modern Optics and Photonics. G. Genty is acknowledged for valuable comments.