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Sub-ppm CO detection in a sub-meter-long hollow-core negative curvature fiber using absorption spectroscopy at 2.3 μm

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ABSTRACT

We demonstrated the sensitive CO detection in a novel hollow-core negative curvature fiber (HC-NCF) with infrared laser absorption at 2.3 μm. The HC-NCF consists of a single ring of eight nontouching silica capillaries around the air core, providing a single-mode light delivery of the 2.3-μm distributed feedback laser. A high coupling efficiency of 90% was achieved with the aid of optimal free-space coupling optics. The hollow-core fiber was used as a gas cell for gas absorption measurement of a total path length of 85 cm. Both direct absorption spectroscopy (DAS) and wavelength modulation spectroscopy (WMS) were adopted to demonstrate the sensor performance by detecting the CO line R(10) at 4297.7 cm^{-1}. In scanned-wavelength DAS, we obtained a minimum detection limit (MDL) of 13 ppm CO, which was limited mainly by the existing mode noise in the HC-NCF. By applying a pressure difference of 0.8 bar between the two ends of the fiber, we demonstrated a very short gas loading time of only 5 s. Finally, we achieved a MDL of 0.4 ppm CO using the WMS technique, corresponding to a noise equivalent absorption of 1.6×10^{-7} cm^{-1}.

1. Introduction

Carbon monoxide (CO), mainly produced by a variety of incomplete combustion activities, is one of the major air pollutants globally. CO even at a low concentration level must be accurately monitored. Gas chromatography (GC) is a traditional technique to measure trace amount of CO [1], but the bulky size and long measurement time (minutes) often limit its further applications. CO sensors based on metal oxide semiconductors have a faster response (< 1 min) and sensitivity at ppm level [2], but they may be influenced by humidity and require a high operating temperature. Recently a new type of CO sensor has been developed to show a high sensitivity with aid of the advanced nanomaterial [3], but it may also be influenced by the ambient species such as O_2.

Tunable diode laser absorption spectroscopy (TDLAS) offers a high sensitivity and fast time-response for trace gas detection. CO has the ro-vibrational absorption bands at 1.55 μm, 2.3 μm and 4.6 μm that can be accessed by commercially available semiconductor lasers. Low-cost telecommunication diode lasers could be used to exploit the overtone and combination bands of CO at 1.55 μm [4–6], but the detection sensitivity is limited by the weak absorption strength at the near-infrared. Sensitive CO sensors could be developed using mid-infrared quantum cascade lasers or interband cascade lasers [7,8], but these commercial mid-infrared lasers are still too expensive to be widely used in actual applications. Hence, sensitive CO detection at 2.3 μm is very promising by implementing a low-cost distributed-feedback (DFB) diode laser that exploits stronger absorption lines than that at 1.55 μm.

Several TDLAS-based CO sensors at 2.3 μm have been reported previously for combustion measurements with an optical path length between a few centimeters and meters [9–11]. In particular, Ebert et al. [9] used a 2.3 μm DFB diode laser to analyze CO in a waste incinerator. A detection limit of 6.5 ppm for an optical path length of 2.56 m was obtained at 1-s acquisition time. To enhance the detection sensitivity, Dang et al. [12] developed a CO sensor for fire alert by using a 14.5-m multipass gas cell. A detection limit of 1.18 ppm was achieved at a 1-s integration time. However, the use of multipass cell in optical gas

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sensors makes the system bulky, heavy and complicated. Additionally, the extra fringe noise caused by the multipass cell significantly deteriorates the detection sensitivity and stability.

Alternatively, hollow-core photonic bandgap fiber (HC-PBF) provides a compact gas cell that confines light-gas interaction in an extremely small volume for a long path length. The HC-PBF normally consists of a periodic cladding structure and an air core in the center of the fiber. Ding et al. [6] reported a CO sensor using a 1.55 μm tunable laser and a 90-cm HC-PBF as the gas cell. A minimum detectable CO concentration of 300 ppm was achieved, which was mainly limited by the large mode noise inside the HC-PBF. Moreover, it took approximately 11 min to load gas samples into the fiber core with an inner diameter of 20 μm. Hence, the two major issues of low sensitivity caused by mode interference and slow gas filling process due to the small hollow core limit the performance of HC-PBF-based gas sensors.

Recently researchers invented a novel type of anti-resonance hollow-core fiber, named hollow-core negative curvature fiber (HC-NCF) [13]. It has an inverted curvature in the core wall that inhibits coupling between the fundamental core mode and the cladding mode, thus leading to single-mode transmission. By introducing a tiny gap between capillaries in the cladding, a node-less structure is created to reduce the overall fiber attenuation in the bending state [14,15]. Compared with conventional HC-PBFs, HC-NCFs usually exhibit multiple transmission bands and have a relatively larger air core.

Although emerged as the cutting-edge research in fiber technology, the use of HC-NCFs for gas sensing has not been fully researched. Nikodem et al. [16] reported CO_2 detection at 2 μm using a 1.35-m HC-NCF and achieved a detection limit of 5 ppm. The HC-NCF has an air core diameter of ˜70 μm that enables a gas loading time of a few seconds. It was observed that higher-order modes could be suppressed in the HC-NCF, which however significantly depended on the coupling condition between the HC-NCF and the pigtailed laser diode. Recently Yao et al. [17] demonstrated a photothermal CO sensor at 2.3 μm using a HC-NCF and achieved a normalized noise equivalent absorption (NNEA) coefficient of 4.4×10^{-8} cm^{-1}WHz$^{-1/2}$.

In this work, we demonstrate the sensitive detection of CO using laser absorption spectroscopy in a HC-NCF. A DFB laser emitting at 2.3 μm is used to access the CO line R(10) at 4297.7 cm^{-1}. The HC-NCF used in this study has an air core diameter of 40 μm and is surrounded by a single ring of eight nontouching capillaries. Compared with the conventional HC-PBF, the HC-NCF provides a broader transmission window covering the $2v_1$ absorption band of CO at 2.3 μm. With the aid of optimized free-space coupling optics, the HC-NCF provides a single-mode and low-loss light delivery at 2.3 μm. Direct absorption spectroscopy and wavelength modulation spectroscopy are both adopted for CO sensing in the hollow-core fiber. The sensor performance such as detection sensitivity, long-term drift, and gas exchange rate are discussed in details.

2. Spectroscopic fundamentals

TDLAS has been widely used for gas sensing applications due to its fast time response and relatively simple sensor configuration. The spectroscopic fundamentals are well documented elsewhere [18] and are hence briefly described here to clarify the notation and units used in this paper.

Generally, when the laser wavelength is scanned across the absorption line of the target gas, i.e., by tuning the injection current of the diode laser, part of the optical power is absorbed by gas molecules. This wavelength-dependent attenuation is related to gas properties such as concentration C, pressure P (atm) and path length L (cm) by Beer-Lambert law:

$$\tau = \frac{I_t}{I_0} = \exp(-C{\cdot}P{\cdot}\chi_v{\cdot}S{\cdot}L), \tag{1}$$

where τ is the fractional transmission defined as the ratio of the transmitted light intensity I_t to the incident light intensity I_0, χ_v (cm) is the line-shape function, and S (cm^{-2} atm^{-1}) is the line-strength of the selected absorption line. The line-shape function χ_v is usually approximated by a Voigt profile that is characterized by the collision-broadened full-width at half maximum (FWHM) and Doppler FWHM.

Additionally, wavelength modulation spectroscopy (WMS), an extension of TDLAS, has been used extensively for the case of weak absorption. In WMS, an additional sinusoidal modulation (at frequency f, kHz) is added to the laser current. For one scan period, the incident laser wavelength $v(t)$ and intensity $I_0(t)$ can be described by the following equations [19]:

$$v(t) = \bar{v}_0 + a\cos(2\pi ft), \tag{2}$$

$$I_0(t) = \bar{I}_0\left[1 + \sum_{m=1}^{\infty} i_m \cos(m{\cdot}2\pi ft + \phi_m)\right], \tag{3}$$

where $\bar{v}_0$ (cm^{-1}) is the center laser wavelength of the laser under modulation, a (cm^{-1}) is the modulation depth, $\bar{I}_0$ is the average laser intensity at $\bar{v}_0$, i_m is the mth Fourier coefficient of the modulation intensity normalized by $\bar{I}_0$, and ϕ_m is the phase shift of the mth-order intensity modulation. Note that laser intensity modulation of most diode lasers could be simply described without considering the higher orders of $m > 2$. The absorption signal with modulation is detected by the photodetector and then demodulated by a lock-in amplifier at its harmonics (2f, 3f, etc.). The second harmonic (2f) signal is of more interest in WMS as it is background free and closely related to absorbance.

3. Experimental

3.1. Single-mode transmission in HC-NCF at 2.3 μm

The 85-cm long HC-NCF used in this work has a core diameter of 40 μm surrounded by a single layer of eight nontouching capillaries shown in Fig. 1. Each capillary has a diameter of 14 μm and a wall thickness of ∼3 μm. Such a novel microstructure forms a negative curvature because the surface normal vector of the core boundary is oppositely directed from the center. This special core boundary keeps the guided fundamental mode away from silica junctions where light attenuation and cladding modes are significant [20]. The node-less cladding structure made of nontouching capillaries forms a free core boundary that eliminates additional optical losses.

When the hollow core fiber (HCF) is used as a gas cell, it is required that the laser beam propagates as a single mode inside the hollow core

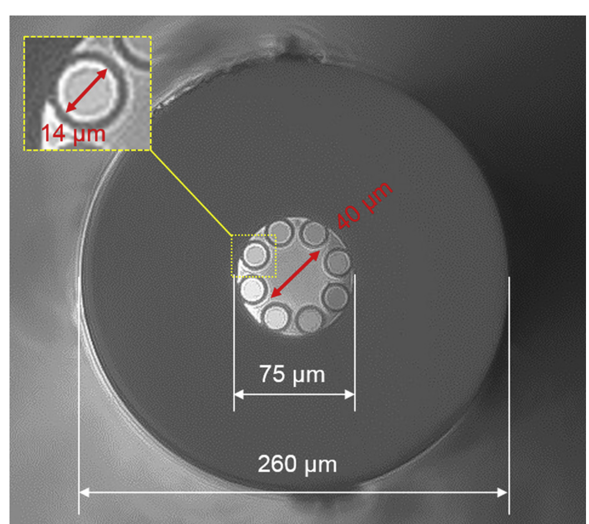

Fig. 1. Node-less cladding structure of the HC-NCF used in this work.

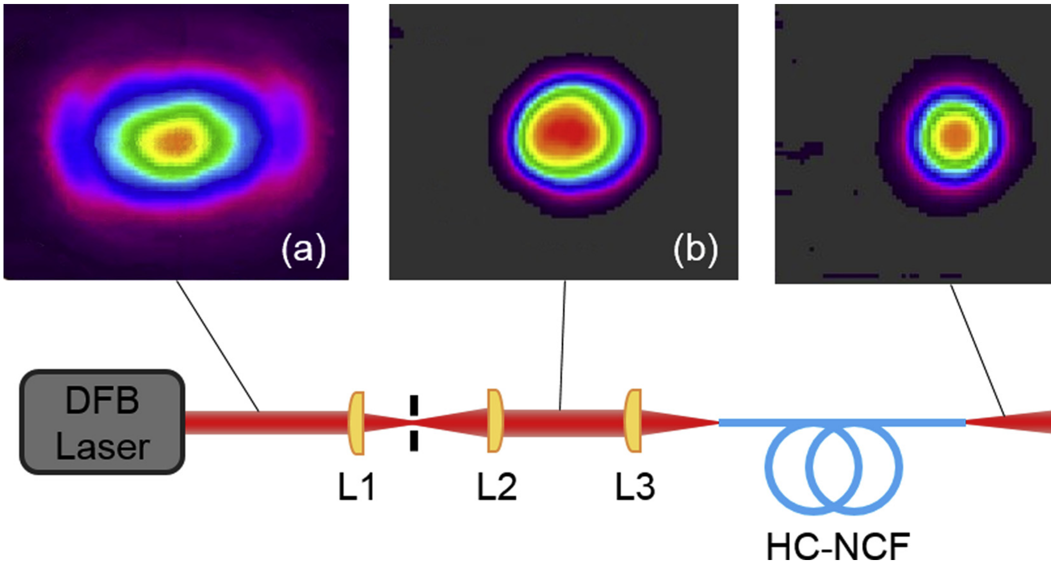

Fig. 2. Beam profiles of the 2.3-μm DFB laser before and after being coupled into the HC-NCF.

to eliminate the mode noise. The recent HC-NCF-based CO_2 sensor adopted the direct butt-coupling between the pigtailed laser diode and the HC-NCF [16]. A few higher modes were excited in the 90-cm long fiber, leading to the evident fringe noise in the acquired signal. In the present work, we coupled the DFB laser with the HC-NCF by free-space optics to achieve nearly single-mode transmission inside the fiber.

A continuous-wave DFB diode laser at 2.3 μm was selected for CO detection as a demonstration. The beam profile of the DFB laser output was captured by a pyroelectric infrared camera, showing an elliptical beam shape as illustrated in Fig. 2(a). To enhance the coupling efficiency, the elliptical laser beam first went through a Keplerian telescope consisting of two convex lenses (f_1 = 20 mm and f_2 = 40 mm); and a pinhole was positioned at the focal point to reshape the beam profile as shown in Fig. 2(b). Then another plano-convex lens (f_3 = 50 mm) was implemented to focus the laser beam into the fiber core. The focal length was selected to produce a spot size of 25 μm which is 0.63 of the core diameter. The spot size was calculated based on the following equation:

$$d = \frac{4\lambda M^2 f}{\pi D}, \tag{4}$$

where d is the focal spot size, M^2 is the beam mode parameter, f is the focal length and D is the input beam diameter located at the lens. By measuring the transmitted laser power, we achieved a coupling efficiency of 90% from the free-space laser beam to the HC-NCF. Fig. 2(c) depicts the perfect Gaussian beam output from the fiber, demonstrating nearly single-mode transmission. We did not see the variation of transmission by bending the fiber to a radius of 10 cm. A detailed investigation could be found in [21] that a low bending loss of 0.2 dB/m at 5-cm bending radius was attained for such HC-NCFs.

3.2. Experimental setup

Fig. 3 depicts the experimental setup of the TDLAS-based CO sensor using the HC-NCF. The DFB laser could be tuned between 4296 cm^{-1} and 4303 cm^{-1} by adjusting the injection current. The target CO line R (10) of the $2v_1$ band at 4297.7 cm^{-1} has an absorption line-strength of 2.937×10^{-21} cm^{-1}/(molecule·cm^{-2}) at room temperature (296 K) [22]. The two fiber ends were cleaved by a ruby scribe to generate clean facets and enclosed in two compact gas cells (4.3 cm^3 in volume) for gas filling. Each of the cells was installed with a CaF_2 window for fiber coupling and optical transmission. The laser beam transmitted through the gas-filled HC-NCF was detected by an infrared photodetector (Vigo Systems).

Both direct absorption spectroscopy (DAS) and wavelength modulation spectroscopy (WMS) were conducted for CO sensing. When performing the DAS measurement, a 10 Hz triangular wave (40 mV_{p-p}) was added to the DFB laser driver (Wavelength Electronics Ltd.) to scan the laser frequency from 4297.1 cm^{-1} to 4298.2 cm^{-1}. The photodetector signal was filtered by an electronic low-pass filter and then acquired by the data acquisition card (DAQ). When performing the WMS measurement, the laser wavelength was dithered at 7 kHz by a sinusoidal waveform and then demodulated at the second harmonic (2f) by a lock-in amplifier. After each TDLAS experiment, a flip mirror was used to direct the laser beam through a germanium etalon for the calibration of laser wavelength [23].

4. Results and discussion

4.1. DAS measurement

Fig. 4(a) presents the measured transmission signal of 0.13% CO in N_2 and the baseline signal of pure N_2. Note that the results shown in Fig. 4 were averaged by 100 scans. All the measurements were conducted at the pressure of 1 bar. Fig. 4(b) depicts the corresponding absorbance along with the profile fitting using the Voigt line-shape function. The x-axis in the figure was converted to the relative frequency by using the germanium etalon. The fitting residual is plotted at the bottom panel of Fig. 4(b). Although the residual is mostly within ± 2% across the absorption feature, a certain fringe noise could be identified that is likely caused by the existing fiber mode noise. The fringe noise in the present work has been minimized by optimizing the coupling optics and laser beam profile as discussed in Section 3.1. With the current optical setup, a signal-to-noise-ratio (SNR) of 99 was obtained for 0.13% CO. Hence, a minimum detection limit (MDL) of 13 ppm CO was estimated for the DAS measurement, corresponding to a noise equivalent absorption (NEA) of 5.2×10^{-6} cm^{-1}.

A longer optical fiber is preferred to achieve better sensitivity and to mitigate the mode interference inside the hollow-core fiber [24]. However, it is normally challenging to load gas samples into the long HC-PBF with a small core diameter of ~10 μm. For instance, it took several hours to fill in a 2-m HC-PBF under free diffusion, which could be reduced to 120 s by creating a pressure difference of 1 bar between the two fiber ends [25]. The slow filling rate significantly increased the sensor response time. To facilitate the gas loading process, microchannels along the side of a HC-PBF were made to achieve a loading

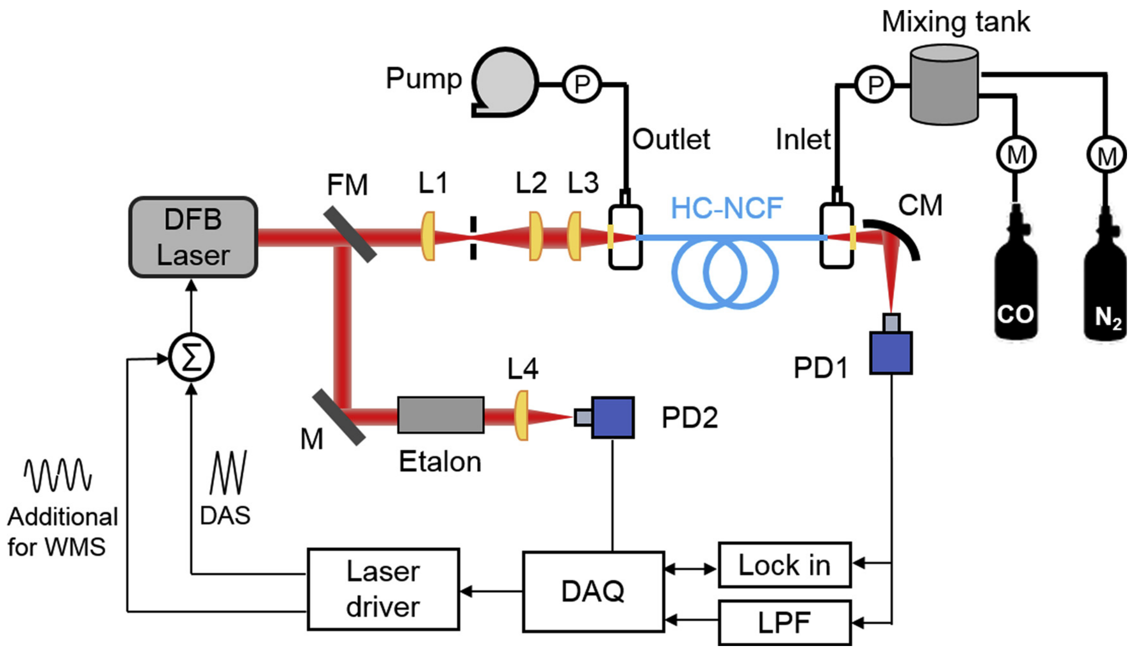

Fig. 3. Experimental setup of the TDLAS-based CO sensor using the HC-NCF. FM: flip mirror; L: lens; M: mirror; CM: concave mirror; PD: photodetector; P: pressure gauge; M: mass flow meter; LPF: low-pass filter.

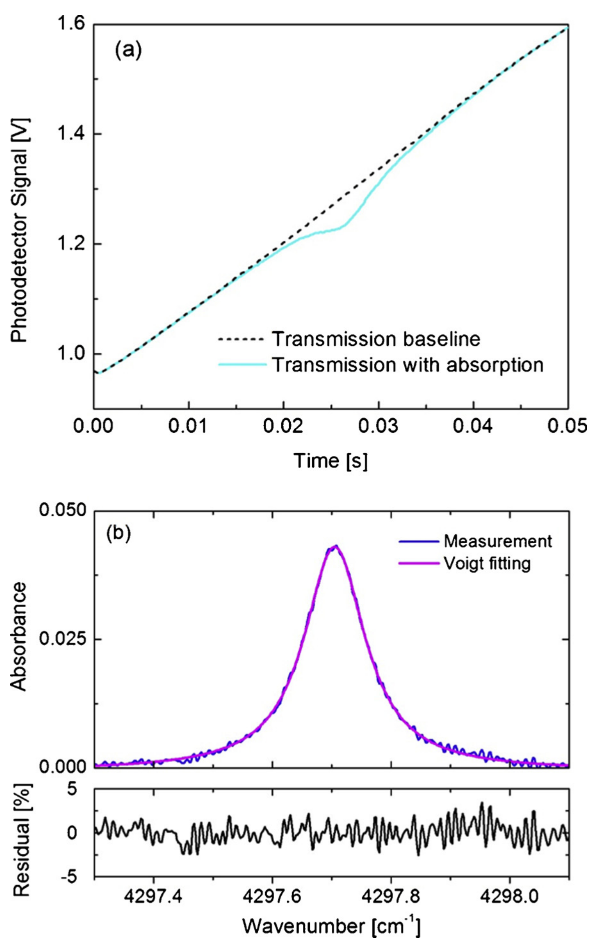

Fig. 4. DAS measurement of 0.13% CO in the HC-NCF. (a) Raw data traces with and without CO absorption; (b) Measured absorbance along with the Voigt fitting.

time of less than 60 s [26]. However, the fabrication of micro-channels on the HC-PBF is complicated and may affect its optical properties. HC-NCFs with exposed-core design have only been investigated theoretically so far [27,28]. For instance, Hao et al. [28] optimized the HC-NCF with an asymmetric gap between the cladding capillaries and a side slot along the fiber.

The HC-NCF used in this work has a relatively larger core diameter of 40 μm. We investigated the gas loading time using the current direct absorption setup. The hollow core was initially filled with N_2 and then evacuated by a vacuum pump. Then the gas sample of 0.8% CO was pressurized into the HC-NCF under a pressure difference of 0.8 bar between the inlet and outlet gas cells (absolute pressure is 1.8 bar and 1 bar, respectively). The DFB laser was swept at 3 Hz and its transmission through the HC-NCF was continually monitored during the gas loading process. Fig. 5 illustrates the measured fractional transmission during the gas filling when the laser wavelength was fixed at the line-center of the CO transition. It is observed that the laser intensity started to drop at 3 s when CO entered the hollow core. After a continuous filling of 5 s, the fractional transmission reached a plateau level,

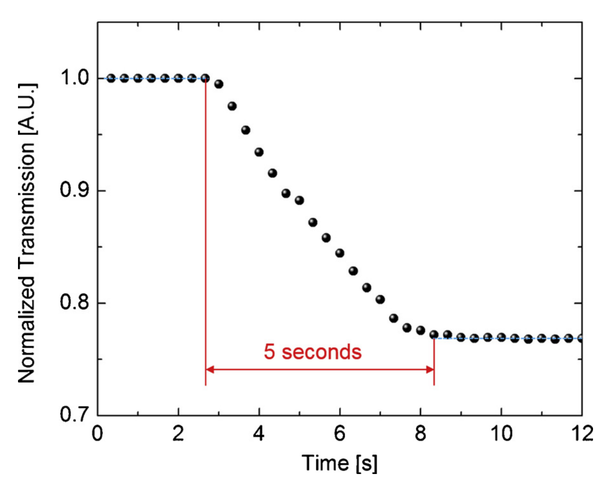

Fig. 5. Laser transmission measurement when loading CO into the HC-NCF at a gauge pressure of 0.8 bar.

indicating the unchanged CO concentration inside the fiber core. Since the laser scanning frequency was relatively high, the sensor response time was mainly determined by the gas loading process. Hence, we obtained a response time of 5 s for such a HC-NCF-based CO sensor with a fiber length of 85 cm.

4.2. WMS measurement

In WMS, the DFB laser wavelength was modulated by a 7 kHz sinusoidal waveform and slowly tuned across the absorption feature by a 1 Hz saw-tooth waveform. The tuning range and target absorption line were selected the same as that used in the DAS measurement. The second harmonic (14 kHz) was then demodulated form the photodetector signal. The modulation frequency of 7 kHz was selected for the current sensor setup because there existed the lowest noise level at the corresponding demodulation frequency of 14 kHz in the system. Again all the measurements were conducted at the pressure of 1 bar.

With the fixed modulation frequency and gas pressure, an optimal modulation depth (see Equation (2)) needs to be selected to maximize WMS-2*f* signal. Here the modulation depth could be adjusted by varying the peak-to-peak voltage of the sinusoidal modulation applied to the laser driver. A gas sample of 0.13% CO/N_2 was filled into the HC-NCF for testing. Fig. 6 depicts the representative WMS-2*f* profiles measured at different modulation depths (in mV). The WMS-2*f* amplitude increases with the modulation depth. However, a non-zero offset in the background also increases due to the unwanted optical fringe in the transmitted light. We thus selected the optimal modulation depth of 25 mV to obtain the relatively strong signal and mitigate the background.

The WMS-2*f* sensor performance was then investigated by measuring CO mixtures with different concentrations inside the HC-NCF. Fig. 7(a) shows two representative WMS-2*f* profiles of two CO concentrations (93 ppm and 1300 ppm). The signal of N_2 is also plotted in Fig. 7(a) to show a standard deviation of 0.009 mV. Fig. 7(b) plots the measured WMS-2*f* amplitudes at different CO concentrations from zero to 0.13%. According to the linear slope of the fitting (0.0068 mV/ppm), we estimated a MDL of 1.3 ppm for the current WMS sensor setup without signal averaging.

The Allan-Werle deviation analysis was performed to investigate the long-term stability of the HC-NCF-based TDLAS sensor. In this test, the gas sample of 0.13% CO/N_2 was pressurized into the hollow core and continuously monitored at the absorption line-center with the results shown in Fig. 8(a). Fig. 8(b) demonstrates the Allan-Werle deviation in units of ppm. The detection limit of the WMS-2*f* CO sensing in the HC-NCF is determined to be 0.4 ppm at 30 s, corresponding to a NEA coefficient of 1.6×10^{-7} cm^{-1}. To our knowledge, it is the first hollow-core photonic crystal fiber-based CO sensor that achieves sub-ppm detection limit.

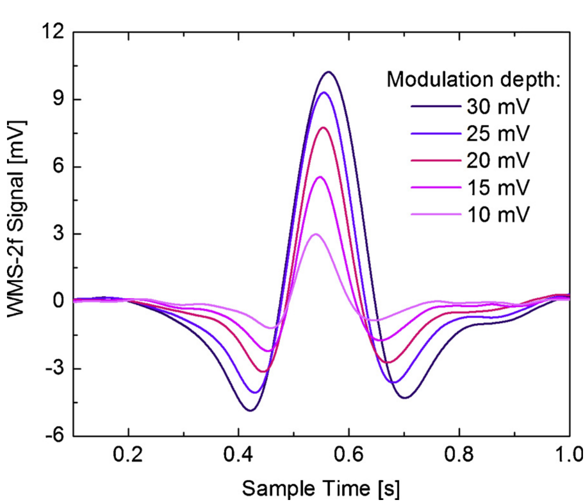

Fig. 6. Measured WMS-2f signals at different modulation depths (in mV).

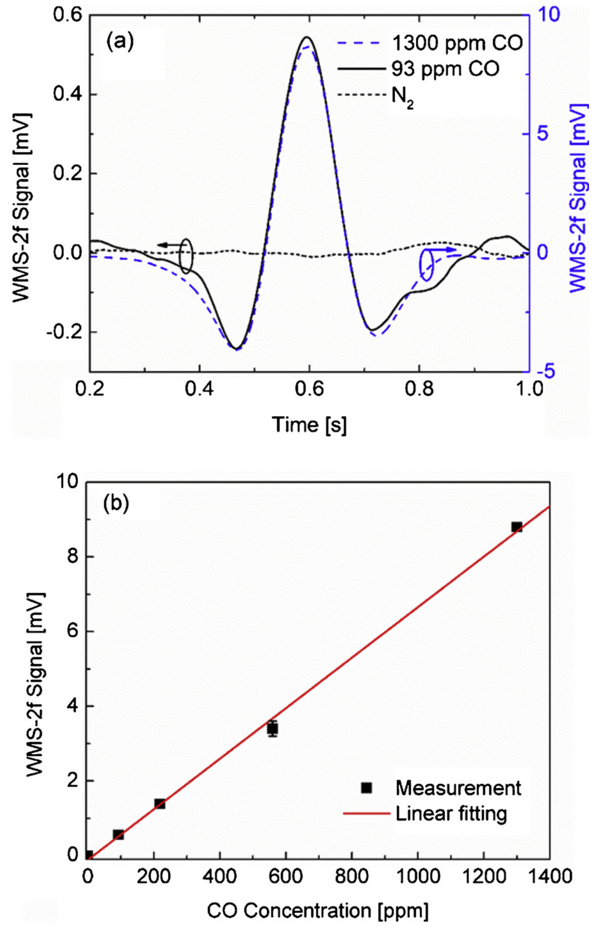

Fig. 7. (a) Representative WMS-2*f* signals of CO/N_2 at two different concentrations; (b) Measured WMS-2*f* amplitude as a function of CO concentration.

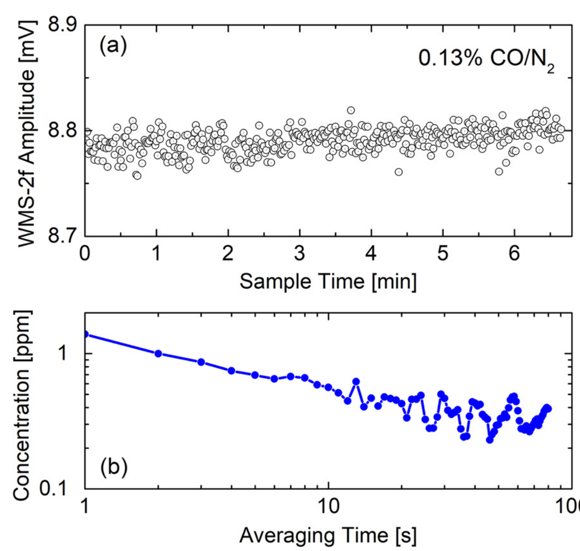

Fig. 8. (a) Continuous WMS-2*f* measurement of 0.13% CO at the absorption line-center; (b) Allan-Werle deviation analysis (in ppm).

5. Conclusions

We reported the sensitive CO detection inside a novel HC-NCF using

TDLAS. The 2.3-μm DBF laser was coupled into the HC-NCF with a high coupling efficiency. Both DAS and WMS techniques were adopted in this work to investigate the HCF-based CO sensor performance. It was verified by DAS that the gas sample could be pressurized (0.8 bar) into the hollow core within 5 s for a fiber length of 85 cm. Meanwhile, a certain fringe noise was observed that is likely caused by the existing fiber mode interference. Finally, we achieved a MDL of 0.4 ppm CO by extending the averaging time of WMS detection to 30 s, corresponding to the NEA of $1.6 \times 10^{-7}\,cm^{-1}$. The detection limit could be further improved by using a longer fiber considering the low transmission loss. Future work will involve the detailed analysis of the bending curvature, bending loss, and fiber length on the sensor performance.

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