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Pulsed photothermal interferometry for high sensitivity gas detection with hollow-core photonic bandgap fibre

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ABSTRACT

Pulsed photothermal interferometry (PTI) gas sensor with hollow-core photonic bandgap fibre (HC-PBF) is demonstrated with a Sagnac interferometer-based phase detection system. Under the condition of constant peak pump power, the optimal pulse duration is found to be $> 1.2 \mu s$ for detecting low-concentration of trace gases in nitrogen, limited by thermal conduction of gases within the hollow-core. Preliminary experiments with a 0.62-m-long HC-PBF gas cell, low peak power ($\sim 20.2 mW$) and a boxcar averager with 10k average times demonstrated a detection limit of 3.3 *p.p.m* acetylene. Detection limit down to ppb or lower is expected with high peak power pump pulses.

Keywords: Optical fibre sensor, photothermal interferometry, gas sensor, photonic crystal fibre, spectroscopy

1. INTRODUCTION

Photothermal interferometry (PTI) is an ultra-sensitive spectroscopic method for gas-and-liquid trace analysis [1]. Instead of measuring the optical spectral attenuation directly, PTI measures the absorption-induced photothermal (PT) phase modulation by the use of an optical interferometer. A pump-and-probe configuration is typically used, in which the absorption of a periodically modulated pump results in modulation of the phase of a probe beam. The use of low-loss hollow-core photonic bandgap fibre for PTI offers important advantages over the free-space systems: (i) a HC-PBF simultaneously confines light and gas sample inside the hollow-core with nearly 100% overlap, enabling strong light-gas interaction over a long distance [2]; (ii) HC-PBF can be coiled to small diameter, enabling more compact sensors; (iii) the PT phase modulation is proportional to the pump light intensity (instead of power) and, for the same pump power level, the light intensity in a HC-PBF can be much higher (2-3 orders of magnitude, because of the much smaller mode-field diameter) than in a free-space beam, hence much larger PTI signal can be achieved. A HC-PBF PTI sensor has been recently demonstrated with a modulated continuous wave (CW) pump and, with $\sim 10 m$ of HC-PBF and $\sim 16 mW$ pump power and we have achieved ultra-sensitivity down to 2 p.p.b (C_2H_2) with a unprecedented dynamic range of nearly six orders of magnitude [3]. These performances are better than any previously reported HC-PBF gas sensors based on the direct absorption spectroscopy.

Here, we report the results of our preliminary investigation on HC-PBF PTI gas sensors with a pulsed pump source. Pulsed lasers with very high peak power in the near infrared wavelength band are now available, which would enable ultra-sensitive gas sensors. We have reported numerical modelling on the dynamics of PT phase modulation within HC-PBF [4], and here we report the results of a detailed experimental investigation on optimizing the modulation parameters and preliminary gas detection experiments. With a pulsed pump with low peak power ($\sim 20.2 mW$), we achieved acetylene gas detection down to 3.3 *p.p.m* with 0.62-m-length HC-PBF and a box-car average with 10 k average times. Detection limit down to ppb level or lower should be achievable with a higher peak power pump and a large number of average times.

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2. EXPERIMENT ON PHASE MODULATION DYNAMICS

The experimental set-up for investigating the phase modulation dynamics of the pulsed PTI gas sensor is shown in Figs.1. An external-cavity laser (ECDL) is utilized as the pump source and after passing through an erbium-doped amplifier (EDFA), it is pulsed in intensity by an acoustic-optic modulator (AOM). The repetition rate of pulse modulation is set to 500 Hz. The sensing HC-PBF is 0.62-m long HC-1550-02 fibre (NKT Photonics) filled with 7500 *p.p.m* in nitrogen. The nominal wavelength of the ECDL is calibrated by a standard gas cell and tuned to the P(9) absorption line of C_2H_2 at 1530.371 *nm*. The PT phase de-modulation is performed by an all-fibre Sagnac interferometer with a 3x3 coupler. A broadband super-luminescent diode (SLED) with 3 dB bandwidth of ~ 41 *nm* and average output power of ~ 10 *mW* is used as our probe beam source. With a 3x3 symmetrical tapered coupler and balanced detection method, the output of the balanced detector (BD) is directly proportional to the phase modulation occurring within the sensing HC-PBF [5]. A 2-km-long single mode fibre (SMF) is used to form the Sagnac loop, giving a loop delay time t_d . For pump pulse duration smaller than the loop delay time t_d , for each of the pump pulses, the output waveform of the system appears as two identically shaped pulses but with reverse signs. The pulses starts to interfere with each other if the pulse duration exceeds the loop delay time t_d .

Figs.2(a) and 2(b) show the output waveforms from the BD for pump pulse durations of 325 *ns* and 2 μs . The experimental waveforms agree well with that predicted with our numerical model [4]. The output waveforms for changing pulse duration from 100 to 2000 *ns* are shown in Fig.2(c). It can be seen from Figs.2(c) that, under the condition of a constant peak pulse pump power level, the peak phase modulation increases approximately linearly for pulse duration of less than ~ 300 *ns*, and keeps increasing before the pulse duration reaches ~ 1.2 μs . There is no significant change in the peak phase modulation for pulse duration larger than ~ 1.2 μs . Hence we may conclude that optical pulse duration for constant peak pump power is ~ 1.2 μs . This result agrees well with our numerical calculation and it was mainly determined by the thermal conduction of N_2 buffer gases filling the hollow-core [4].

To determine the magnitude of PT phase modulation, we used a piezoelectric (PZT) phase modulator to calibrate the phase modulation. The phase modulator is constructed by coiling several-meters of SMF around the PZT, and is connected into the Sagnac loop. A sinusoidal signal with a modulation frequency of 50 kHz is used to drive the PZT to generate a π (peak amplitude, observed in oscilloscope) phase difference between the clockwise (CW) and counter-clock-wise (CCW) waves. And assuming that phase modulation is linearly proportional to the amplitude of voltage applied on PZT, we can generate a 0.5 rad phase changes by reducing the voltage applied to the PZT and the output waveform can be observed in oscilloscope. The magnitude of the pulsed PT phase modulation can then be estimated by comparing the pulsed output waveform (Figs.2(c)) with the amplitude of the sinusoidal waveform corresponding the 0.5 rad phase modulation. The peak phase modulation for different pulse duration is determined by the above method and shown in Figs.2(d). The maximum peak phase modulation for pulse duration > 1.2 μs is determined to be 0.047 rad, which is close to the predicted value 0.069 rad in our numerical model in [4]. The measured normalized phase modulation coefficient is $1.204 \times 10^{-6} \text{ rad} \cdot \text{ppm}^{-1} \cdot \text{mW}^{-1} \cdot \text{m}^{-1}$.

3. GAS DETECTION AND ABSORPTION LINESHAPE MEASUREMENT

Gas detection experiments were conducted with the same setup shown in Figs.1 except that the oscilloscope is replaced by a boxcar averager (SRS250). The sensitivity of boxcar is 1V/5mV and the gate width is about 300 *ns*. In our experiments, we choose the pulse duration of the pump beam to be 3 μs which satisfies the condition of maximum phase modulation and also avoids the overlap of output waveform. Figs.3(a) shows the output from the Boxcar with average times of 10 and 10000 with nominal wavelength of pump beam fixed at the centre of the absorption line. The signal levels remain almost the same while the amplitude fluctuation (indicated as 1σ noise level) is significant reduced with a larger number of average times. And the signal-to-noise ratio (SNR) appears to be proportional to the square root of average times N as shown in Figs.3(b). It indicates that our system is mainly limited by white noise [6] and a better sensitivity could be achieved with larger number of average times. With average times of 10000, the amplitude of signal output from the boxcar is about 3.3 *mV* and 1σ noise level is about 0.0015 *mV*. The SNR is about 2247. The lower detection limit in terms of noise equivalent gas concentration can then be estimated to be 3.3 *p.p.m* for a SNR of unity.

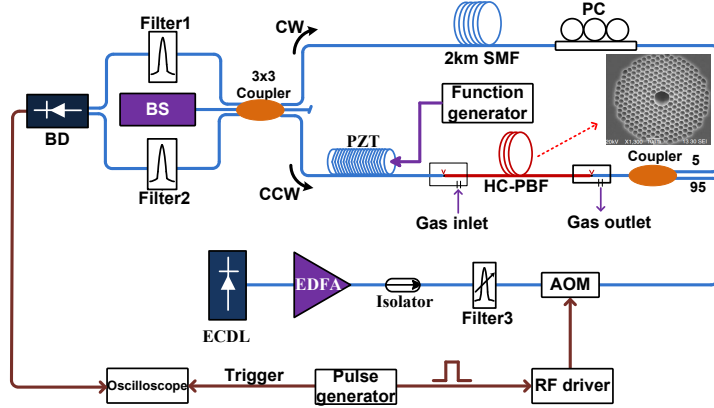


Figure 1. Experimental set-up for gas detection with 0.62-m-long HC-PBF. An external-cavity diode laser (ECDL) with wavelength around 1530.371 nm is used as pump beam. The tunable filter-3 filters out the noise accompanying the EDFA. The fixed wavelength filters (filter-1 and filter-2, both have center wavelength of 1553.33 nm and 3-dB bandwidth of 1 nm) are used to filter out the residual pump. AOM: acoustic-optic modulator, BS: broadband source (probe), PC: polarization controller, BD: balanced-detector. The output of BD is connected to an oscilloscope to observe the dynamics of the photothermal signal. A piezoelectric-transducer (PZT) is used for phase calibration purpose. The inset figure is the cross-sectional image of the sensing fibre (NKT Photonics HC-1550-02 fibre) that has a core diameter of $\sim 11\text{ }\mu\text{m}$.

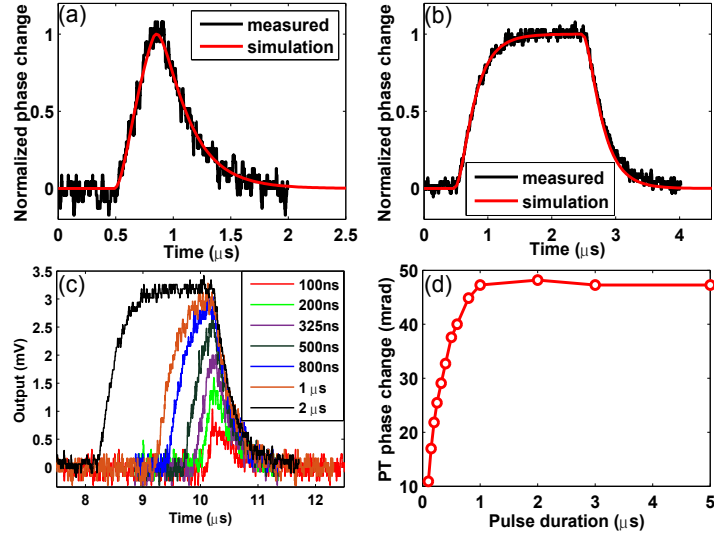


Figure 2. Comparison between output waveforms of BD and computed results with pump pulse duration of (a) 325 ns, (b) $2\text{ }\mu\text{s}$. (c) Output waveforms of BD and (d) peak PT phase modulation for different pulse durations of the pump beam.

For the fixed pump pulse duration of $3\text{ }\mu\text{s}$, the peak amplitude of the PT phase modulation with increased peak power level of the pump pulses are measured and plotted in Figs.3(c). A good linear relationship is obtained. Obviously, the detection limit could be improved with increasing pump power and number of averaging times. We speculate that for the same system parameters but a peak pump power of a few Watts, which is not difficult to achieve, p.p.b. level of acetylene is possible.

The absorption spectral shape of absorption line can also be determined by the use of our PTI system. Figs.3(d) shows the boxcar output signals when the wavelength of the pump beam is tuned from 1530.10 to 1530.47 nm (corresponding to the wavenumber range of $6533.94\text{ cm}^{-1} \sim 6535.52\text{ cm}^{-1}$). The signals are normalized against the maximum PT signal at the absorption line center. By fitting the data to a Lorentz line shape, we obtained a half width half maximum (HWHM) of 0.09757 cm^{-1} , quite close to that obtained from HITRAN data base (0.0820 cm^{-1}) [7].

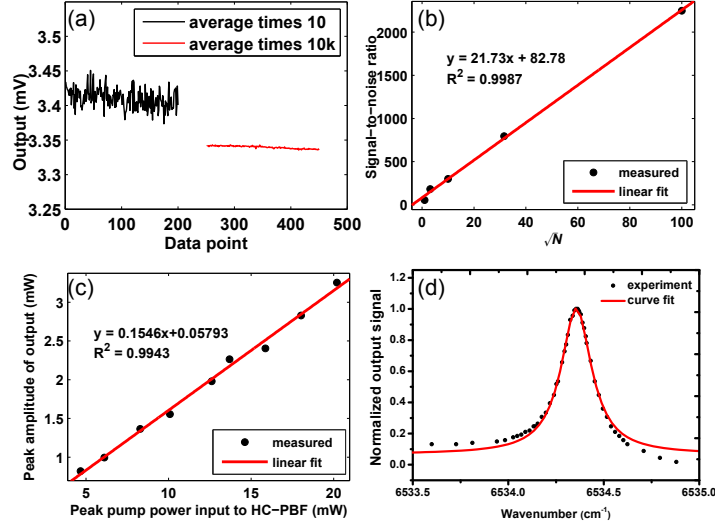


Figure 3. Results of gas detection experiments with HC-1550-02 fibre filled with 7500 *p.p.m* C_2H_2 in nitrogen. (a) Boxcar output with different number of average times. (b) SNR of the output as function of averaging times N . (c) Output from balanced detector (BD) for different pump peak power delivered to the HC-PBF. (d) The spectral shape of absorption line measured with the PTI system.

4. CONCLUSION

We presented the results of gas detection with pulsed all-fibre PTI with a HC-PBF gas cell. Experiments shows that, for constant pump power, there exists optimal pulse durations to achieve maximum PT phase modulation. For low-concentration of acetylene in nitrogen, the pulse duration should be $> 1.2 \mu s$. Preliminary gas detection experiments achieved a minimum detectable gas concentration of 3.3 *p.p.m* of C_2H_2 with 0.62-m-length HC-PBF, low-peak power of pump pulses of 20.2 *mW* and a boxcar average with 10k average times. Further significant improvement in detection sensitivity is expected by using pump pulses with a higher peak power and a larger number of averages.

5. ACKNOWLEDGMENTS

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