



All-optical light-induced thermoacoustic spectroscopy for remote and non-contact gas sensing

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ABSTRACT

All-optical light-induced thermoacoustic spectroscopy (AO-LITS) is reported for the first time for highly sensitive and selective gas sensing, in which a commercial standard quartz tuning fork (QTF) is employed as a photo-thermal detector. The vibration of the QTF was measured by the highly sensitive fiber-optic Fabry-Pérot (FP) interferometry (FPI) technique, instead of the piezoelectric detection in the conventional LITS. To improve the stability of the sensor system, a compact QTF-based fiber-optic FPI module is fabricated by 3D printing technique and a dual-wavelength demodulation method with the ellipse-fitting differential-cross-multiplication algorithm (DW-EF-DCM) is exploited for the FPI measurement. The all-optical detection scheme has the advantages of remote detection and immunity to electromagnetic interference. A minimum detection limit (MDL) of 422 ppb was achieved for hydrogen sulfide (H₂S), which was ~ 3 times lower than a conventional electrical LITS sensor system. The AO-LITS can provide a promising approach for remote and non-contact gas sensing in the whole infrared spectral region.

1. Introduction

Quartz tuning fork (QTF) as a resonant acoustic-electric transducer based on its piezoelectric property has been successfully applied in trace gas photoacoustic sensing since 2002, which is called quartz-enhanced photoacoustic spectroscopy (QEPAS) [1–8]. The QTF has the advantages of a high resonance frequency, a high quality factor, a small size and a low cost, which enable the QEPAS sensor to have the immunity to ambient acoustic noise and the capability to analyze ultra-small volume gas samples with respect to the conventional PAS sensor based on a photoacoustic cell and a microphone [9–23]. However, the QTF required to be immersed in the target gas in the QEPAS sensor, so that the QTF can be corroded. Thus, the performance of the QTF can be deteriorated as detecting the corrosive gas for a long time, which limits the application of the QEPAS sensor in some harsh environments such as

combustion field.

Light-induced thermoacoustic spectroscopy (LITS) as a laser-absorption-spectroscopy-based gas sensing approach was first reported in 2018 [24], which used a QTF as a photothermal detector. Different from measuring acoustic signal generated by gas absorption of light energy in a QEPAS sensor, the QTF is exploited to measure the absorption variation of light intensity in a LITS sensor, and the light beam is directed on the QTF's surface instead of between the gap of QTF's prongs. The thermal energy generated by light absorption is transformed into the mechanical vibration of the QTF by the light-thermo-elastic conversion, and subsequently the mechanical vibration is enhanced due to the resonant characteristic of the QTF [24–30]. The mechanical vibration of the QTF can also generate acoustic waves and be detected [31]. The LITS sensor system is similar to that of tunable diode laser absorption spectroscopy (TDLAS), except that the commercial

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photodetector is replaced by the QTF. Therefore, the LITS sensor can realize the non-contact measurement in contrast to the conventional PAS/QEPAS sensor. Compared with the conventional commercial photodetector-based TDLAS technique, the QTF-based LITS technique has the merits of low cost, small size, high sensitivity, large power dynamic range and signal enhancement by increasing of the excitation laser power, and in particular it can achieve the trace gas detection in the whole infrared spectral region, even THz spectral region, without requiring any cooling system [24–34]. However, the conventional piezoelectric measurement is vulnerable to electromagnetic interference and difficult to achieve long-distance remote detection.

For toxic and corrosive gas (e.g. H_2S) detection, the remote and non-contact measurement is expected. In some specific fields (e.g. power industry), the gas sensor also required to be immune to severe electromagnetic interference. In fact, fiber-optics sensing technique based on Fabry-Pérot interferometry (FPI) structure, can offer a safe and effective approach to remotely measure the vibration of the QTF not depending on its piezoelectric effect, thus effectively avoiding electromagnetic interference. For the FPI detection, there are several algorithms. The conventional quadrature working point (Q-point) demodulation method can be affected by the instability of Q-point and the dynamic range is limited by the linear region of interference spectrum [35–38]. The white-light-based phase demodulation method cannot demodulate the high-frequency signal (such as ~ 32 kHz for the QTF) [14,39,40]. Therefore, the dual-wavelength demodulation method with the ellipse-fitting differential-cross-multiplication algorithm (DW-EF-DCM) is exploited to measure the vibration of the QTF in this work, which has the advantages of good stability, strong robustness, large demodulation frequency range and large dynamic range, and which is not affected by the instability of Q-point [41–43].

In this paper, we develop an all-optical LITS (AO-LITS) sensor to detect H_2S for the first time. The LITS technique is used to provide the capability of non-contact measurement. The vibration of the QTF produced by the light-induced thermo-elastic effect is measured by the fiber-optic FPI technique instead of the conventional piezoelectric detection. A 1576.29 nm distributed feedback (DFB) laser is used as an excitation light source for the H_2S detection. A Herriot multipass gas cell (MGC) with an effective optical pathlength of 3.3 m is used to enhance the light absorption of the H_2S gas. To improve the stability and practicability of the sensor system, a QTF-based integrated fiber-optic FPI module is designed and fabricated by means of the 3D printing technique. The DW-EF-DCM demodulation method was innovatively applied in the developed AO-LITS sensor system. The fiber-optic interferometry technique makes the sensor system have the abilities of remote detection and immunity to electromagnetic interference. Such an AO-LITS sensor system can also be suitable for trace gas remote detection in the mid-infrared, far-infrared and even THz spectral regions since the quartz crystal has an inherent light absorption in a broad spectral region and the probe laser of the fiber-optic FPI technique is independent of the excitation light for gas absorption.

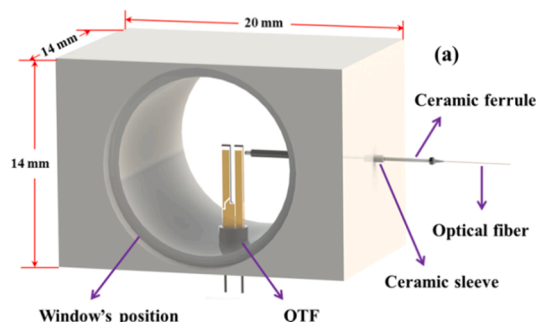


Fig. 1. (a) 3D schematic diagram of the QTF-based fiber-optic FPI module (b) Physical photograph of the FPI module.

2. Theory and design of AO-LITS sensor

2.1. QTF-based fiber-optic FPI module

In a QTF-based AO-LITS sensor, the information of target gas concentration can be obtained by measuring the vibration of the QTF. A commercial standard QTF and the fiber-optic FPI probe were integrated to a compact module using 3D printing technique. Fig. 1(a) and (b) show the 3D schematic diagram and physical photograph of the module, respectively. The module with dimensions of $20\text{ mm} \times 14\text{ mm} \times 14\text{ mm}$ consisted of two same interchangeable windows, a commercial standard QTF, a ceramic ferrule, a ceramic sleeve and an optical fiber. The two same interchangeable windows with a diameter of 12.7 mm were installed on the front and back of the QTF, respectively, which was employed to insulate the external environment and allow access to the laser beam. The resonance frequency and the quality factor of the used QTF at atmospheric pressure were experimentally determined to be 32,776.2 Hz and 10,283.5, respectively. The ceramic ferrule was employed to fix the optical fiber. The end surface of the optical fiber and the silver-coated surface of the QTF's prong formed a low-finesse F-P cavity. The ceramic sleeve was used to assist in fixing the ceramic ferrule. All these parts were fixed and sealed by Torr Seal Epoxy.

2.2. DW-EF-DCM demodulation method based on QTF vibration

Fig. 2 shows the schematic diagram of a FPI detection unit for QTF vibration measurement based on dual-wavelength demodulation technique. A near-infrared tunable laser source (Alnair Labs, TLG-210) was employed as the probe light source to generate two probe laser beams with the different wavelengths around 1550 nm. Multiplexing and demultiplexing of the two probe laser beams were achieved by means of

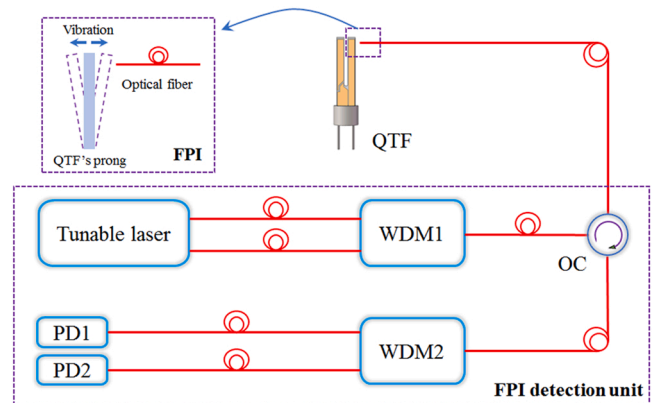
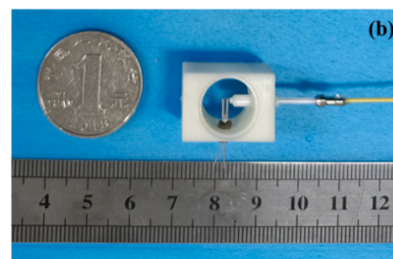


Fig. 2. FPI detection unit for QTF vibration measurement based on dual-wavelength demodulation technique: WDM, wavelength division multiplexer; OC, optical circulator; PD, photodetector.



two wavelength division multiplexers. A fiber-coupled optical circulator was used to separate incident light and reflected light for the QTF-based F-P cavity. The interferometric signals of the two probe laser beams were detected by two commercial fiber-coupled photodetectors (PDs), respectively. All the components of the dual-wavelength demodulation system were connected using the optical fibers. The output signals of the two PDs were recorded by a data acquisition (DAQ) card (National Instrument, PCIe-6376).

In the QTF-based F-P cavity, the vibration of the QTF's prong causes the F-P cavity length to oscillate. The signal obtained by each PD can be described as [41–43]:

$$V_1 = A_1 + B_1 \cos\left[\frac{4\pi nL}{\lambda_1} + \varphi(t)\right] \quad (1)$$

$$V_2 = A_2 + B_2 \cos\left[\frac{4\pi nL}{\lambda_2} + \varphi(t)\right] \quad (2)$$

where V_1 and V_2 represent the output signals of PD1 and PD2, respectively. A_1 , A_2 , B_1 and B_2 are constant coefficients determined by the probe laser and the PDs, L is the F-P cavity length, n is the refractive index of air in the F-P cavity, λ_1 and λ_2 are the two probe laser wavelengths, respectively, $\varphi(t)$ is the phase signal of the QTF vibration, t is time. The phase difference of the two probe laser wavelengths can be expressed as:

$$\Delta\varphi = \frac{4\pi nL}{\lambda_1} - \frac{4\pi nL}{\lambda_2} \quad (3)$$

when $\Delta\varphi \dagger k\pi$ (k is a integer), there is an elliptical relationship between V_1 and V_2 . The A_1 , A_2 , B_1 , B_2 and $\Delta\varphi$ can be obtained by ellipse fitting. The output signals of the two PDs can be normalized by eliminating direct current component and alternating current amplitude. The normalized signals of V_1 and V_2 can be expressed as:

$$V_{1n} = (V_1 - A_1)/B_1 = \cos\left[\frac{4\pi nL}{\lambda_1} + \varphi(t)\right] \quad (4)$$

$$V_{2n} = (V_2 - A_2)/B_2 = \cos\left[\frac{4\pi nL}{\lambda_2} + \varphi(t)\right] \quad (5)$$

The phase signal of the QTF vibration can be calculated by using DCM algorithm:

$$\varphi(t) = \frac{\int (V'_{1n} V_{2n} - V_{1n} V'_{2n}) dt}{\sin(\Delta\varphi)} \quad (6)$$

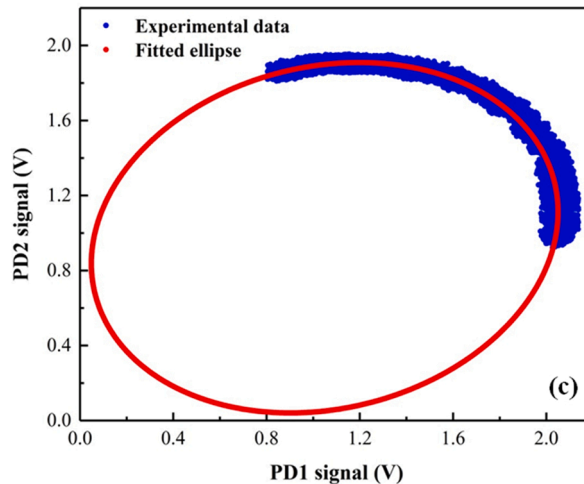
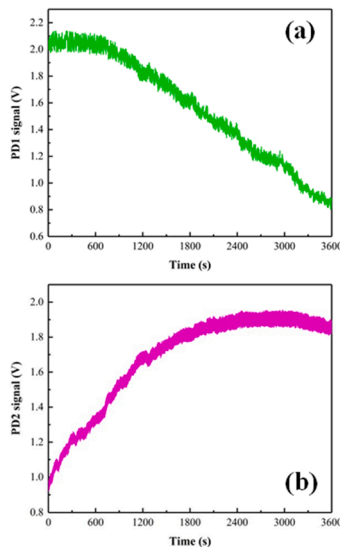


Fig. 3. (a) Output signals of PD1 (b) Output signals of PD2 (c) Ellipse fitting for the combined signals of PD1 and PD2.

where V'_{1n} and V'_{2n} are the derivatives of V_1 and V_2 , respectively. The vibration amplitude of the QTF related to the oscillation amplitude of the F-P cavity length, can be obtained by demodulating $\varphi(t)$ at 32,776.2 Hz (the resonance frequency of the used QTF) using a lock-in amplifier.

To obtain the fitted ellipse of the QTF-based F-P cavity, the FPI module and detection unit ran for one hour. Affected by the environmental parameters such as temperature and humidity, the refractive index of air in the cavity drifted, resulting in the changes of the signals of the two PDs, which is plotted in Fig. 3(a) and (b), respectively. With the PD1 and PD2 signals as the abscissa and ordinate, respectively, an ellipse was achieved as shown in Fig. 3(c). After an ellipse fitting, A_1 , B_1 , A_2 , B_2 and $\Delta\varphi$ were achieved to be 1.0493, 1.0008, 0.9751, 0.9342 and 0.4530π , respectively.

3. Experimental results and discussions

3.1. Experiment setup and optimization

H_2S as a toxic and corrosive gas was chosen as the analyte due to the widely detection demand in environmental monitoring, chemical production and power industry [44–50]. Fig. 4 shows the experimental setup of the AO-LITS sensor system for H_2S detection. A continuous wave DFB fiber-coupled diode laser with an output power of ~ 20 mW

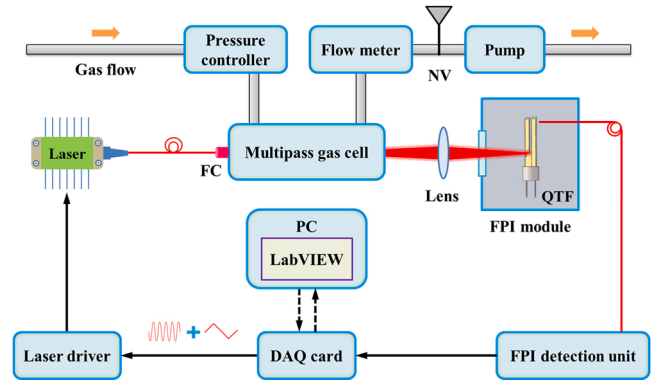


Fig. 4. Experiment setup of the AO-LITS sensor system: FC, fiber-coupled collimator; NV, needle valve; PC, personal computer.

was selected as an excitation light source to detect H_2S at 1576.29 nm, where has an interference-free H_2S absorption line [51]. The laser operating temperature and current were controlled by an integrated laser driver (Wavelength Electronics, LDTC0520). Wavelength modulation spectroscopy (WMS) and second-harmonic ($2f$) detection technique were exploited to improve the signal-to-noise ratio (SNR). A triangular signal plus a sinusoidal signal of 16,388.1 Hz (half of the resonance frequency of the used QTF) were generated by a LabVIEW controlled DAQ card to scan and modulate the laser current, respectively, for $2f$ signal detection. The output laser was collimated by a fiber-coupled collimator, and then injected into a compact Herriot MGC (Healthy Photon, HPHC-B). The MGC has an effective optical pathlength of 3.3 m with 33 optical passes to enhance light absorption of H_2S gas. The output laser emitted from the MGC was focused to the bottom of the QTF's prongs by a flat-convex focusing lens with a 30 mm focal length, which is the optimum laser focal point for LITS proved in Ref. [25]. The positions of the MGC and the QTF-based FPI module were adjusted using a high precision three-axis translation stage. The pressure in the QTF-based FPI module was atmospheric pressure, which avoided the use of any pressure control unit. A LabVIEW-based lock-in amplifier (LIA) demodulated the phase signals from the FPI detection unit in $2f$ mode at 32,776.2 Hz. The time constant and the filter slope of the LIA were set to 300 ms and 12 dB/oct, respectively, corresponding to an equivalent noise bandwidth (ENBW) of 0.416 Hz. All data were recorded and processed by a LabVIEW software program in a personal computer.

Since H_2S is toxic, the MGC uses a closed structure with a gas inlet and outlet. The pressure in the MGC was maintained at atmospheric pressure by a mini diaphragm pump (KNF Technology, N813.5ANE) and a pressure controller (MKS Instruments, 649B13TS1M22M). A needle valve and a mass flow meter (Alicat Scientific, M-2SLPM-D/5M) were used to control the gas flow rate at 150 mL/min. A 50 ppm $\text{H}_2\text{S}/\text{N}_2$ standard gas was diluted with pure N_2 to produce $\text{H}_2\text{S}/\text{N}_2$ mixtures with different H_2S concentration levels using a gas dilution system (MCQ Instruments, GB100). For field applications, the MGC can be open. In this case, no gas control unit is required. In addition, the AO-LITS can also be applied in an open environment (without the gas cell).

To improve the AO-LITS signal amplitude, the laser wavelength modulation depth was optimized. The MGC was filled with 50 ppm $\text{H}_2\text{S}/\text{N}_2$ gas mixture and operated at atmospheric pressure. The laser wavelength was tuned to the peak of the target H_2S absorption line. The 16,388.1 Hz sinusoidal signal was used to modulate the laser driving current with the modulation depth from 4 mA to 18 mA. Fig. 5 shows the measured AO-LITS signal amplitudes under different modulation depths. According to the results, the modulation depth was set at 12 mA to obtain the strongest AO-LITS signal amplitude in the following

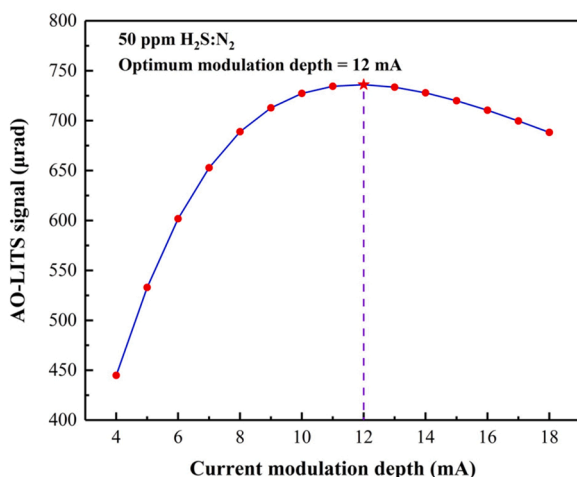


Fig. 5. Signal amplitude as a function of current modulation depth.

investigations.

3.2. Performance evaluation of the AO-LITS sensor system

The measured $2f$ spectrum of 50 ppm H_2S (solid line) at atmospheric pressure was shown in Fig. 6(a). The noise level of the AO-LITS sensor system was evaluated. Pure N_2 was flushed into the MGC. The measured noise was depicted in Fig. 6(b). As a comparison, a conventional electrical LITS (E-LITS) scheme [25] was implemented with the same experimental conditions. The measured $2f$ spectrum of 50 ppm H_2S and noise of pure N_2 by piezoelectric detection were plotted in Fig. 6(a) (dash line) and Fig. 6(c), respectively. It can be found that the SNR of AO-LITS is ~ 3 times stronger than that of E-LITS.

The linearity of the concentration response of the sensor system was evaluated by five different H_2S concentration levels ranging from 10 ppm to 50 ppm. The laser wavelength was tuned to the peak of the target H_2S absorption line to obtain the $2f$ signal amplitude. Sixty data points of the $2f$ signal amplitudes were recorded continuously with a 1 s data acquisition time at each H_2S concentration level, as shown in Fig. 7 (a). The average values of the measured results for each step as a function of the H_2S concentrations were plotted in Fig. 7(b). It confirms the linear response of the AO-LITS sensor system to H_2S concentrations by a linear fitting with a R-square value of > 0.999 . Based on the slope of the fitting curve and 1σ noise of pure N_2 , a H_2S minimum detection limit (MDL) of 422 ppb was achieved ($\text{MDL} = 1\sigma \text{ noise/slope}$) [52]. The noise equivalent absorption (NEA) coefficient and the normalized noise equivalent absorption (NNEA) coefficient can be calculated by the following equations [4,25]:

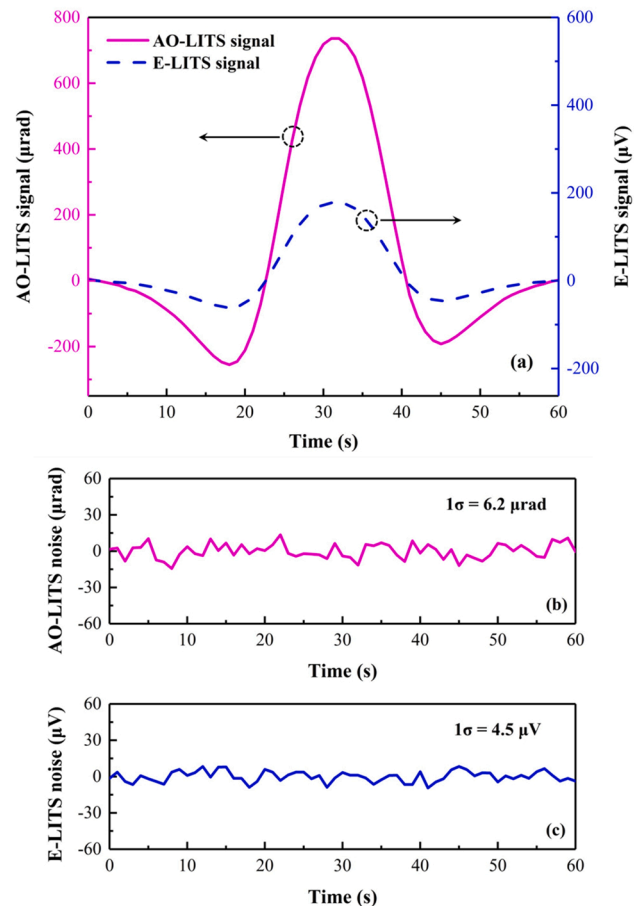


Fig. 6. (a) $2f$ spectrum measured by AO-LITS (solid line) and E-LITS (dash line) (b) AO-LITS noise level (c) E-LITS noise level.

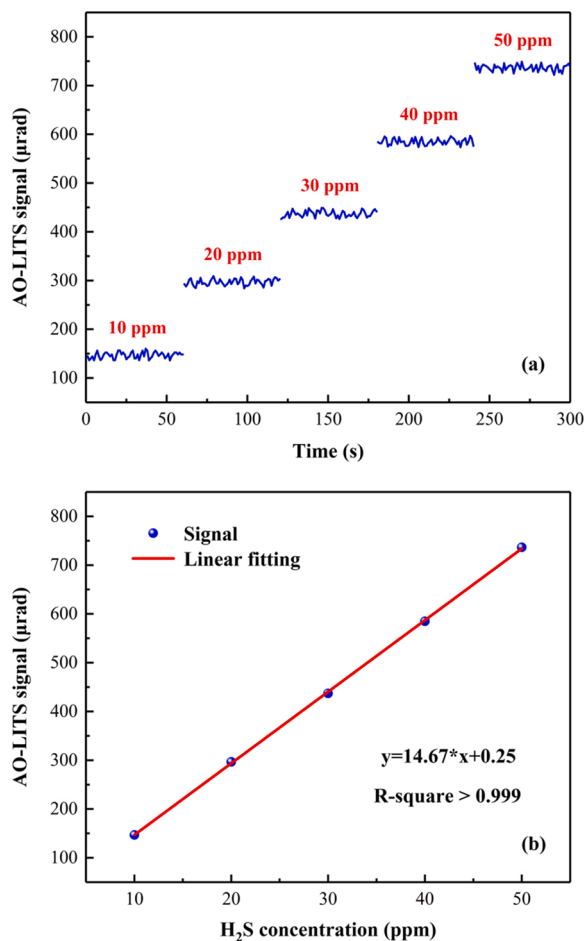


Fig. 7. (a) AO-LITS signals at the different H₂S concentration levels (b) Response linearity of the AO-LITS sensor system.

$$NEA = \frac{\alpha_{min}}{\sqrt{ENBW}} \quad (7)$$

$$NNEA = P \bullet NEA \quad (8)$$

α_{min} is the minimum detectable absorption coefficient, which can be calculated to be $1.5 \times 10^{-8} \text{ cm}^{-1}$ by the HITRAN database [51]. ENBW is the equivalent noise bandwidth of the LIA, which is 0.416 Hz, when the time constant and the filter slope of the LIA were set to 300 ms and 12 dB/oct, respectively. P is the excitation laser power of ~ 20 mW. Hence, the NEA coefficient and the NNEA coefficient were determined to be $2.3 \times 10^{-8} \text{ cm}^{-1}/\sqrt{\text{Hz}}$ and $4.6 \times 10^{-10} \text{ W}\cdot\text{cm}^{-1}/\sqrt{\text{Hz}}$, respectively.

3.3. Long-time stability of the AO-LITS sensor system

To verify the long-time stability of the developed AO-LITS sensor system, the 50 ppm H₂S was continuously measured for one hour. The laser wavelength was tuned to the peak of the target H₂S absorption line. The measurement results are shown in the Fig. 8 (red line). Since the dual-wavelength demodulation system can degrade into the conventional Q-point demodulation system when only one wavelength is used, we measured the 50 ppm H₂S using the conventional Q-point demodulation method, as shown in Fig. 8 (blue line). It can be observed that when the operating time exceeded ~ 1000 s, the signal began to drop, which meant that the working point drifted (not at Q-point). However, the signals remained stable for the DW-EF-DCM demodulation method. The experimental results confirmed that the developed AO-LITS gas sensor based on the DW-EF-DCM demodulation method has a good stability for long-time gas detection.

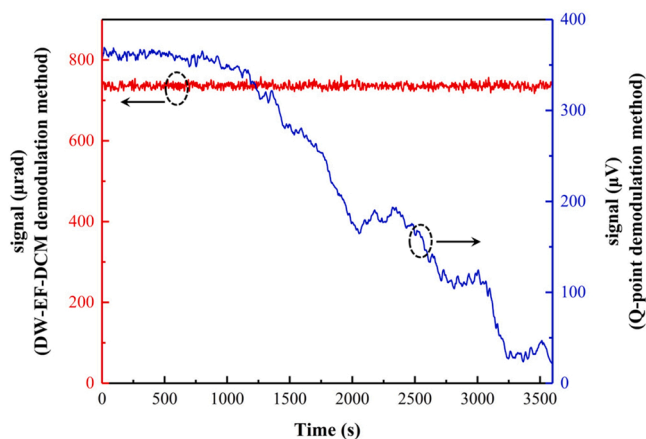


Fig. 8. Comparison of DW-EF-DCM demodulation method (red line) and Q-point demodulation method (blue line) for the AO-LITS sensor system.

4. Conclusions

In this work, a novel AO-LITS gas sensor system based on fiber-optic FPI measurement was reported by using the DW-EF-DCM demodulation method. A compact QTF-based fiber-optic FPI module was designed and the vibration of the QTF was measured by the FPI technique. At atmospheric pressure, the MDL for H₂S detection is 422 ppb, which is ~ 3 times better than that of the conventional electrical LITS sensor system. Since the QTF does not need to be in contact with the target gas, the AO-LITS can realize non-contact measurement and avoid the interference of gas flow noise on highly-sensitive optical detection [13,14]. The detection sensitivity can be further improved by decreasing the operating pressure in the QTF-based FPI module (to obtain a higher quality factor of the QTF) and/or increasing the effective optical pathlength [25, 53]. With respect to E-LITS, AO-LITS removes the restriction on the use of piezoelectric materials. In addition, the DW-EF-DCM demodulation method has large dynamic range for vibration measurement. As a result, with the same geometry of tuning fork, the materials with a higher laser absorption coefficient (such as black phosphorus and perovskite) and with a lower Young's modulus (such as KBr and KCl) could offer higher sensitivity [54–56]. On the other hand, since the DW-EF-DCM algorithm can be affected by the instability of the probe laser power in the case of continuous operation for a very long time (such as several days), the optimization of the demodulation algorithm for FPI measurement will also be our next research work, in order to further improve the stability and calibration-free ability of the AO-LITS sensor system.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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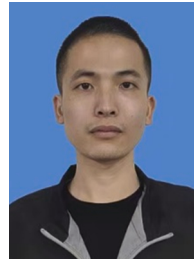
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