Palm-sized methane TDLAS sensor based on a mini-multi-pass cell and a quartz tuning fork as a thermal detector

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Abstract: A palm-sized methane (CH\textsubscript{4}) tunable diode laser absorption spectroscopy (TDLAS) sensor is reported, in which a quartz tuning fork (QTF) is used as a thermal detector, working together with a mini-multi-pass cell (mini-MPC) to compose a gas detection module (GDM) with a compact dimension of 78 mm × 40 mm × 40 mm. A 1.65 \textmu m near-infrared distributed feedback (DFB) laser is installed in the sensor for CH\textsubscript{4} detection. A minimum detection limit (MDL) of 52 ppb is achieved at an integration time of 300 ms, corresponding to a normalized noise equivalent absorption coefficient (NNEA) of 2.1×10\textsuperscript{-8} cm\textsuperscript{-1} W/Hz\textsuperscript{1/2}. A seven-day continuous monitoring of atmospheric CH\textsubscript{4} concentration is implemented to verify the sensor’s long-term stability.

1. Introduction

Methane (CH\textsubscript{4}) as a clean energy gas is widely distributed in nature, which is not only one of the main components of natural gas, methane gas and mine gas, but also an important greenhouse gas in the atmosphere [1–5]. Since the 21st century, many studies have shown that the greenhouse effect follows the same shift trend as atmospheric CH\textsubscript{4} concentration, that is, CH\textsubscript{4} gradually becomes one of the important gas components of the intensified greenhouse effect on the surface of the earth. Although the concentration of CH\textsubscript{4} in the atmosphere is much lower than that of carbon dioxide (CO\textsubscript{2}), its superior infrared absorption capacity results in the fact that a greenhouse effect of CH\textsubscript{4} is 25 times greater than that of CO\textsubscript{2} [4,5]. The sources of atmospheric CH\textsubscript{4} are mainly divided into natural sources (marshes, wetlands...) and CH\textsubscript{4} emissions caused by human activities (agriculture, waste, the extraction and burning of oil and natural gas...). Anthropogenic CH\textsubscript{4} emissions mainly include the extraction and burning of fuel, agricultural activities (livestock...), and waste disposal. The unregulated anthropogenic CH\textsubscript{4} emissions are the main cause of the rise in atmospheric CH\textsubscript{4} concentration. Therefore, the real-time and in-situ monitoring of atmospheric CH\textsubscript{4} concentration becomes very important and urgent, that means that a compact, robust, real-time, and online CH\textsubscript{4} gas sensor is an immediate need.

In recent years, trace gas sensors based on optical methods have been researched and applied in various industries due to the advantage of fast response, non-invasive, superior selectivity, and real-time detection [3,5–28]. Direct absorption spectroscopy (DAS) has attracted many researchers’ interest for its advantages of simple operation, high stability, and detection sensitivity independent of gas relaxation rate. Wavelength modulation spectroscopy (WMS) technique due to its efficient background suppression ability is applied to DAS to improve the detection sensitivity of the sensor, which is called tunable diode laser absorption spectroscopy (TDLAS). TDLAS technology has been widely applied in respiratory diagnosis, greenhouse gas detection, gas leakage and other fields in combination with excitation light sources in different “fingerprint”
spectral regions [3, 5, 15–17]. For example, a highly sensitive CH$_4$ sensor with a minimum detection limit (MDL) of 10 ppb using a dense-pattern multi-pass cell (MPC) is reported in 2016 [15]. A CH$_4$ sensor with a minimum detection limit of 117 ppb based on three-dimensional (3D) printed miniature fiber-coupled MPC was reported in 2020 [3].

The core device of TDLAS technology is a MPC equipped with a photodetector. For laser absorption at different spectral regions, it is usually necessary to equip a proper photodetector with corresponding response wavelength, such as a Si photodetector for visible region, an InGaAs photodetector for near-infrared region, a HgCdTe photodetector for mid-infrared region. With the development of the semiconductor industry in the past 20 years, the output power of commercial semiconductor lasers is increasing steadily, and >1 W semiconductor lasers are readily available commercially. However, conventional photodetectors cannot bear a high incident light power due to its small dynamic range, and thus the TDLAS sensing performance cannot benefit from the development of the semiconductor industry. As a result, the characteristics of photodetectors including wavelength selection and small dynamic range limit the applications of the TDLAS.

Quartz tuning forks (QTFs) known as wrist watch crystals are mainly used for time control in watches, clocks and nowadays in electronic toys and household appliances as time reference. Their forked structure working together with quartz piezoelectric effect forms a microforce transducer, which was verified to have a detection capacity of femto-newton [29] and which has been employed for the detections of sound wave [18], thermal wave [21] and even atomic force [30]. In 2018, a QTF was selected as a thermal detector in TDLAS technology [22], which has a potential to avoid the wavelength selectivity of conventional photodetectors and to take advantage of high-power excitation light sources. However, it is a big challenge to design such a practical TDLAS sensor based on the QTF-thermal detector because 1) A MPC with absorption path length on the order of 10 m has a typical physical length of ~30 cm and volume of 250 ml, which is an unsatisfactory match for a small size QTF and semiconductor laser source; 2) A complicated optical system is required to focus a laser beam on the assigned position of a QTF surface after the beam exits from the MPC, which makes the sensor system complex, non-robust and bulky.

In this manuscript, we report on the development of a palm-sized, robust, and sensitive CH$_4$ sensor based on a fiber coupled mini-MPC and a QTF-thermal detector with beam control. All optical parts are integrated into a so-called gas detection module (GDM), which address the issues of the MPC volume mismatch and the complicated beam control. The reliability of the palm-sized CH$_4$ sensor is finally verified via a seven-day continuous monitoring of atmospheric CH$_4$ concentration, using a 1.65 µm near-infrared distributed feedback (DFB) laser as an excitation source.

2. Experimental apparatus

The basic principle of the QTF-thermal detector is as follows: the modulated laser interacting with gas molecules radiates to the surface of the QTF, and then the QTF absorbs the laser energy to generate heat energy, which can be converted into the mechanical vibration of the QTF based on the light induced thermoelastic effect [22, 23]. The mechanical vibration can be converted into electrical signals because of the piezoelectric effect of quartz crystal. Hence the target gas concentration information can be obtained through the acquisition and processing of electrical signals.

The CH$_4$ sensor was built as shown in Fig. 1(a). The overall set up consisted of three parts: control electronics unit (CEU), fiber coupled GDM and signal acquisition and processing unit. A 1.65 µm DFB diode laser (NTT Electronics, Inc., NLK1U5EAAA) was installed on the butterfly-encapsulated laser base inside the CEU as the excitation light source, and the temperature and current of the laser were driven through the circuit board inside the CEU. The function generator embedded in CEU was controlled by LABVIEW software in laptop to output the ramp wave for signal scanning and the sinusoidal wave for signal modulation. The tail fiber
of the DFB laser was connected to the FC/APC interface of the GDM, which was composed of a fiber coupled mini-MPC and a QTF-thermal detector. The laser beam in the mini-MPC, that interacted with the target gas molecules, was focused on the QTF surface through a flat convex lens. The output electrical signal of the QTF was amplified by the transimpedance amplifier, and then transmitted to the signal acquisition and processing unit, which consisted of a lock-in amplifier (Stanford Research Systems, Inc., SR830) and a laptop. A QTF can only respond to laser radiation whose modulation frequency is equal to the resonance frequency of the QTF. But the application of the second harmonic technology led to the half of the resonance frequency used. The lock-in amplifier was set at a $2f$ demodulation mode. The modulation frequency of the laser was used as the synchronous signal of the lock-in amplifier to demodulate the electrical signal from the transimpedance amplifier, and then the demodulated electrical signal was processed by LAVIEW software in the laptop to obtain the concentration information of the target gas.

![Figure 1](image)

**Fig. 1.** (a) Schematic diagram of the CH$_4$ sensor; (b) Photograph of the GDM; (c) Photograph of the QTF-thermal detector.

A photograph of the palm-sized GDM is shown in Fig. 1(b). The fiber collimator was composed of a 1.5 $\mu$m single mode fiber and a G-lens. One end of the fiber collimator was FC/APC interface, which was used to connect the tail fiber of the laser, and the other end was a collimating beam output port with the G-lens, which outputs Gaussian beam with a $\sim$600 $\mu$m waist diameter at the working distance of 80 mm. A mini-MPC based on two one-inch spherical mirrors with $>99.06\%$ high reflectivity and a 25-mm curvature radius was used in GDM. Under the condition of 39.4-mm spherical mirror spacing, the optical path of laser interaction with gas molecules was 4.2 m due to the optimal laser pass times of 107 formed by seven-nonintersecting-circle spot pattern on each spherical mirror. The detailed simulation and design of this type of MPCs can be found in our previous papers [3,24,25]. A 2-mm diameter hole with a specific tilt angle was drilled on
two spherical mirrors in the MPC by precision machining technology as the laser inlet and outlet. The whole MPC, which mainly consists of a cage structure, used the open design to complete the detection of atmospheric CH\textsubscript{4} concentration. Two cage plates were made by 3D printing technology as the bracket of the spherical mirrors. The material of the GDM based on the 3D printing technology is resin, which is allowed to operate at a temperature <46 °C. The cage plate of the incident spherical mirror with a fiber collimator bracket was designed to integrate the fiber collimator with the MPC. A QTF-thermal detector was placed behind the cage plate of the exit spherical mirror. The positions of the QTF-thermal detector and the MPC were adjusted through a XYZ Translation Stage, so that the exit laser can radiate on the optimal excitation position of the QTF surface [22,23]. The fiber collimator, the MPC and the QTF-thermal detector were fixed by Torr seal with a dimension of 78 mm × 40 mm × 40 mm.

The QTF-thermal detector consisting of a flat convex lens, a QTF and an enclosure was assembled as shown in Fig. 1(c). A flat convex CaF\textsubscript{2} lens with a diameter of 8 mm and a focal length of 11 mm was used to focus the exit laser beam from the MPC into a ~100-µm spot. The commercial standard QTF with a size of 3 × 8 mm was sealed into the enclosure as the thermal detector. The resonance frequency and the quality factor of the QTF at atmospheric pressure were 327,803.5 Hz and 10,797, respectively, which were obtained by analyzing the QTF frequency response curve by use of the electric excitation method [28]. Since the QTF-thermal detector exhibits the higher detection sensitivity under low pressure conditions [23,26], a Teflon tubing with an outer diameter of 3 mm was used to connect the enclosure to a pressure indicator (MKS Instruments, 662C13TDE) and a vacuum pump (Oerlikon Leybold Vacuum Inc., D16C), monitoring the pressure and providing a negative pressure environment, respectively. The QTF was placed at the focal length of the flat convex lens, and the focused laser beam hit the optimal excitation position of the QTF by adjusting the position of the QTF [22,23]. The enclosure of the QTF-thermal detector with a dimension of 12 mm × 13 mm × 18 mm was made by 3D printing technology.

3. Results and discussion

An interference-free CH\textsubscript{4} absorption line located at 6046.95 cm\textsuperscript{-1} with an absorption intensity of 1.455 × 10\textsuperscript{-21} cm/molecule was selected to implement the detection of the atmospheric CH\textsubscript{4}. The laser temperature and current were set at 16.38 °C and 130.5 mA, respectively. The corresponding output power of the laser was 22.86 mW, while the laser power after passing through the MPC was reduced to 2.4 mW due to the reflection loss of the spherical mirrors in the MPC. The scanning current range of the ramp wave from the function generator was between 119 - 149 mA, which covers the complete CH\textsubscript{4} characteristic absorption line. Moreover, the amplitude of the sinusoidal wave from the function generator was optimized to 9.5 mA. The time constant and filter slope of the lock-in amplifier were set at 300 ms and 12 dB/oct, respectively, corresponding to a detection bandwidth of 0.833 Hz. Furthermore, the 3f locking frequency technology was applied to lock the laser wavelength onto the peak of the CH\textsubscript{4} absorption line for ensuring the stability of the laser output power and wavelength [18].

To optimize the performance of the CH\textsubscript{4} sensor, the GDM was placed in an acrylic gas chamber. The signal response ability of a QTF as thermal detector is positively correlated with the accumulation time of the QTF, which is directly affected by the quality factor of the QTF [26]. The higher quality factor of the QTF can be achieved via reducing the pressure of the enclosure. The resonance frequency and quality factor of the QTF at 8 Torr were determined to be 327,809 Hz and 27,205, respectively. The quality factor is 2.5 times higher than that at 700 Torr. The response ability of the QTF-thermal detector at 700 Torr and at 8 Torr was assessed by comparing the signal amplitudes of 5 ppm CH\textsubscript{4} at normal atmosphere, as shown in Fig. 2. The results show that the signal peak value obtained by the QTF-thermal detector at 8 Torr is 2.6 times higher than at 700 Torr, indicating that the quality factor plays a crucial role in the QTF.
response ability. Therefore, the pressure in the QTF-thermal detector was maintained at 8 Torr to obtain an optimal sensitivity of the CH₄ sensor.

The linear relationship between the CH₄ concentrations and the 2f signal amplitudes was evaluated by measuring the CH₄ concentrations in the range of 30 - 2 ppm, as shown in Fig. 3(a). The standard 30-ppm CH₄ gas and the high-purity nitrogen are mixed to generate different CH₄ concentrations using the gas mixer. A R-squared value of >0.9999 was obtained by linearly fitting the signals in different CH₄ concentrations, which indicates that the CH₄ sensor has the capability of an excellent linear output. The 2f signal of the 2 ppm CH₄ was collected and shown in Fig. 3(b), and the peak value of the signal is 298 µV. The 1σ noise level without gas absorption was calculated to be 7.8 µV, resulting in a signal-to-noise ratio (SNR) of 38. An MDL of 52 ppb for the CH₄ sensor was achieved, which yielded a normalized noise equivalent absorption coefficient (NNEA) of 2.1 × 10⁻⁸ cm⁻¹ W/Hz¹/². The above results demonstrate a higher sensitivity than the reported CH₄ photodetector-based sensor with an MDL of 117 ppb [3]. As shown in Fig. 4, the stability and accuracy of the CH₄ sensor were assessed by the Allan variance method, which was based on long-term measurements of the 5 ppm CH₄ signal. The curve of Allan variance decayed with the trend of 1/√t during the average time before 200 s, and an MDL of 0.94 ppb was obtained when the average time reaches 200s.

A seven-day continuous monitoring of atmospheric CH₄ concentration was implemented from Nov. 2 to Nov. 8, 2020. The CH₄ sensor was placed on the third floor of the Shaw Amenities Building on the Shanxi University campus in Taiyuan, China with GPS coordinates: 37°47′58.1″N 112°35′14.2″E. The palm-sized open GDM was put out the window. The measurement time of one data point was 6.4 s, while the average value of the CH₄ concentrations within 10 minutes was stored to save the storage space of the hard disk. As shown in Fig. 5(a), the average value of atmospheric CH₄ concentration in seven days is 2.4 ppm, and the concentration fluctuation range is between 1.7 - 3.6 ppm. During Nov. 2 and Nov. 4, it can be clearly observed that the highest CH₄ concentration in a day occurred at 2:30 am and the lowest concentration occurred at 4:00 pm. However, the change of CH₄ concentration was relatively irregular between Nov. 5 and Nov. 8. Especially on Nov. 5, the CH₄ concentration rose from 8:00 am and peaked at 12:00 am, and then continued to decline until 5:00 pm. However, in terms of the overall trend of the CH₄ concentration over these four days, the maximum CH₄ concentration of the day occurred at 2:30 am and the lowest at 8:30 am. The above irregular behavior is due to the CH₄ disorganized emission from the nearby natural gas station and the influence of wind speed and
Fig. 3. (a) Signal amplitudes at different CH$_4$ concentrations and linear fitting result; (b) 2f spectrum of 2 ppm CH$_4$.

Fig. 4. Allan variance analysis for the CH$_4$ sensor.

direction. Figure 5(b) shows the diurnal profile of atmospheric CH$_4$ concentration in seven days. The results show that the maximum and minimum of the CH$_4$ concentration in one day occur at 3:00 am and 1:00 pm respectively, which is consistent with the previous reports [3].

Fig. 5. (a) Real-time monitoring results of atmospheric CH$_4$ concentration on the Shanxi University campus from Nov. 2 to Nov. 8, 2020; (b) Diurnal profile of atmospheric CH$_4$ concentration in seven days.
4. Conclusion

In conclusion, we developed a CH\textsubscript{4} sensor based on a mini-MPC and a QTF-thermal detector, and demonstrated its effective ability to detect atmospheric CH\textsubscript{4} concentration. Using a QTF as a thermal detector, the QTF does not need to directly contact with gas molecules, which prevents the QTF from being corroded by some gas molecules during long-term contact. Moreover, the broad absorption characteristic of quartz avoids the photodetector replacement. The characteristics of the small size and high-quality factor of the QTF lead to the fact that QTF-thermal detectors are more portable and have a potential of higher detection sensitivity than conventional photodetectors. The highly integrated design makes the CH\textsubscript{4} sensors more compact and more convenient. Future research would focus on applying the mini-MPC and the QTF-thermal detector to the mid-infrared spectral region in order to achieve the higher detection sensitivity.

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