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Continuous trace gas concentration measurement based on quartz-enhanced photoacoustic spectroscopy technique

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A continuous trace gas concentration measurement using quartz-enhanced photoacoustic spectroscopy technique based on harmonic detection and LabVIEW program was demonstrated in this paper. The wavelength of the laser was locked at the absorption line peak with the help of the third harmonic signal. Using a 2.33 μm continuous wave, distributed feedback fiber-coupled diode laser, a long-term monitoring of carbon monoxide concentration for 85 s was performed to demonstrate reliable operation of the quartz-enhanced photoacoustic spectroscopy sensor.

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1. Introduction

Quartz-enhanced photoacoustic spectroscopy (QEPAS) technique was firstly reported in 2002 as an innovation of microphone-based photoacoustic spectroscopy [1]. QEPAS technique utilizes a tiny piezoelectric quartz tuning fork (QTF) as the sensing device to detect the acoustic wave. QTF is a commercial available, millimeter sized detection element with high Q-factor (~10000 at one standard atmospheric pressure, ~100000 in vacuum) and narrow resonant frequency band (~ 4 Hz), which resulted in an excellent immunity to environment noise [2-6]. Usually in QEPAS, a QTF with resonant frequency f_0 of ~32.76 kHz acts as an acoustic transducer [7-10]. Therefore, the corresponding energy accumulation time ($t=Q/f_0$) is ~300 ms, which is significantly longer than any photoacoustic cell used in traditional photoacoustic spectroscopy (PAS) [11]. Due to the merits of high sensitivity, fast response and small volume, QEPAS technique has been applied in many trace gases detection [12-21].

Usually a wavelength modulation spectroscopy (WMS) with 2nd harmonic detection is utilized in QEPAS for sensitive gas concentration measurement. Modulation of the laser current is performed by applying a sinusoidal dither to the direct current ramp of the laser at half of the QTF resonance frequency ($f = f_0/2$ ~ 16.3 kHz) [22]. This process is called scanning mode for QEPAS measurement, which means that the laser wavelength is tuning to cover the gas absorption line. Usually the frequency of the scanning (ramp) is lower than 1 Hz. As a result, this limits the fast response of QEPAS sensor. Therefore, for some toxic and dangerous gases, continuous measurement is needed.

Carbon monoxide (CO) is a hazardous gas. Excessive exposure to CO can cause deprivation of oxygen in human tissue [23]. Hence, a sensitive and continuous detection of CO has an important significance. In this paper, a continuous CO-QEPAS detection based on harmonic detection and LabVIEW program was reported. By using the third harmonic ($3f$) signal resulted from the reference cell and the digital proportion integration differentiation (PID) technique, the laser wavelength is locked at the peak of the gas absorption line. In this way, wavelength shift of the laser was well avoided and the gas concentration was detected stably.

2. Theoretical analysis

2.1. Wavelength modulation spectroscopy technique

From the Lambert-Beer Law, the laser intensity is given by:

$$I(v) = I_0(v)\exp[-\alpha(v)L] \quad (1)$$

where $I_0(v)$ is input intensity of the laser, $I(v)$ is the output intensity, $\alpha(v)$ is absorption coefficient at frequency v, L is the effective absorption length. Modulation of the laser current in WMS was controlled by applying a sinusoidal dither to the direct current ramp at the half of the QTF resonant frequency. Then the instantaneous frequency of output laser can be described as:

$$v(t) = v_0 + a\cos(2\pi ft) \quad (2)$$

when the absorption coefficient was very small, the difference between input and output laser intensity can be ignored in principle. With $\alpha(v)$ expressed in Fourier series, the output laser intensity can be descripted by the relation as follows:

$$I(v) = I_0 - I_0 L \sum_{n=0}^{\infty} A_n \cos(2n\pi\, ft) \qquad (3)$$

where A_n is the Fourier coefficient. The lock-in amplifier was used to analyze different orders harmonic component. Fig. 1 depicts the forms of harmonic component at the orders from one to four. Since the extremum of even order located at the peak of absorption line, the even order harmonic component can be used to invert gas concentration. Due to the fact that the harmonic component reduces with the growth of the order, the second harmonic (2*f*) can be chosen as the optimum object. During the process of wavelength scanning and modulation, odd harmonic components display two extremums successively. Therefore, the emission wavelength of the laser can be locked at the zero point between the two extremums and in this condition the laser wavelength coincide with the absorption line. With the help of PID feedback circuit, the real-time, continuous gas detection can be achieved.

2.2. LabVIEW program

This section mainly describes how the programming tool LabVIEW control the gas detection system. In LabVIEW, graphical programming language (G language) was used in the form of procedure charts to program instead of characters in C language. The virtual instrument software architecture (VISA) is a standard for configuring, programming, and troubleshooting instrumentation systems comprising serial or USB interfaces. VISA provides the programming interface between the hardware and development environments. The LabVIEW program for QEPAS sensor contains two parts, which are: (1) Front panel: data setting and displayer; (2) Program block diagram: connecting line, front panel icons, functions, data structure, sub VIs (VI: Virtual instruments; sub VI: the VI within another VI). A serial port connector as the hardware is controlled by LabVIEW to delivery data.

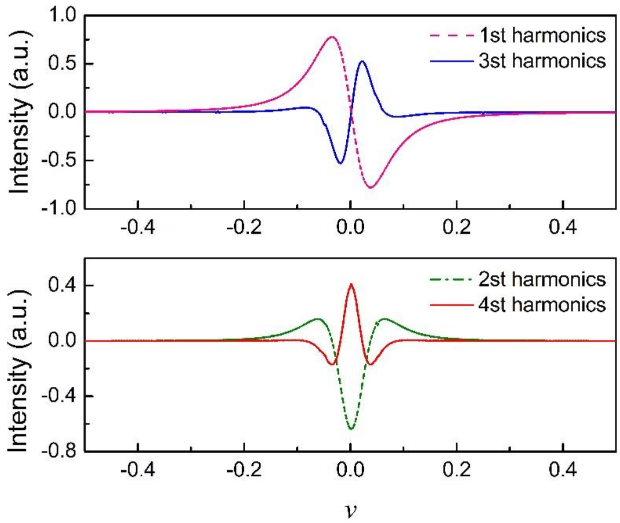

Fig. 1. Harmonics signal with different orders: (a) odd harmonics; (b) even harmonics

Fig. 2 shows the systematic process from the collection of data to the display of continuous concentration measurement.

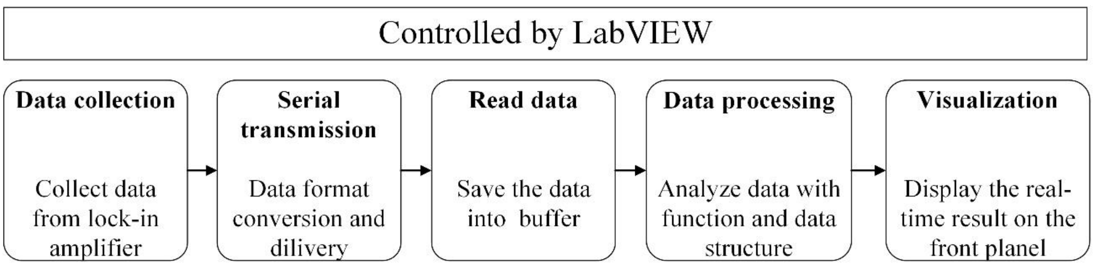

Fig. 2. Flow diagram of QEPAS signal analysis process

1) Data collection: Data from the data acquisition card, which is demodulating from lock-in amplifier.

2) Serial transmission: Digital interface is performed via RS232 serial port possessing the parameters of 115200 baud, 8 bits, no parity, 1 stop bit, and hardware flow control. The serial port will realize the reception of parallel data characters from CPU, the transformation from parallel data characters into continuous serial ones, then send them to computer.

3) Data reading: The VISA write control and read control play an important section in this part. The buffer of VISA can be regarded as a save area whose size can be set by specific control and instruction.

4) Data processing: In this section, the data wanted would be picked out and analyzed by functions, data structure, iteration or other methods.

5) Visualization: Finally, the harmonic signals and the calculated concentration will be displayed on the front

panel.

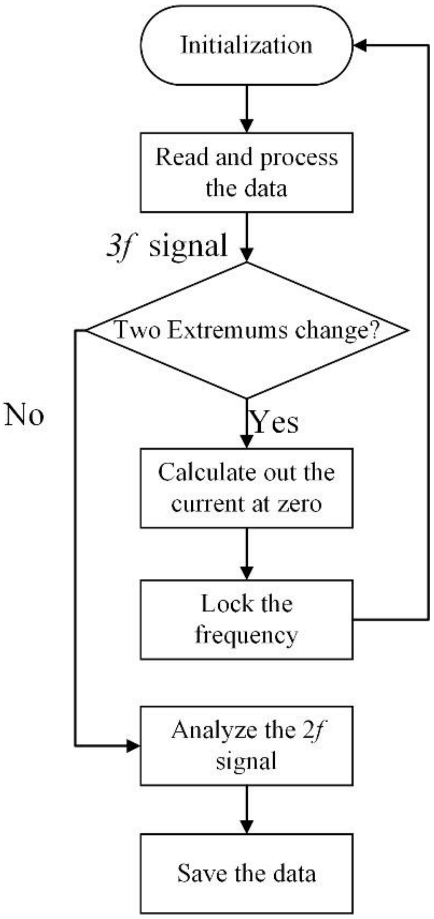

Fig. 3. The flow diagram of locking laser wavelength based on third harmonic signal

The demodulation of 2*f* signal in the QTF and 3*f* signal in the reference cell were performed by the lock-in amplifier. The feedback of the laser was realized using general-purpose interface bus (GPIB) to control the current source (Mode No. LDC-3724, Newport) of the diode laser. Therefore, the locking of the laser wavelength was achieved. Fig. 3 displays the locking procedure of 3*f* signal in the LabVIEW program. The locking of wavelength is judged by a statement whether two extreme value of 3*f* signal change. If the extreme values change, a wavelength shift happened which was caused by the fluctuation of laser temperature. A recalibration, representing as calculating the zero point of 3*f* signal, occurs to ensure the accuracy of locked laser wavelength.

3. Experimental setup

The experimental configuration of CO detection was depicted in Fig. 4. The excitation laser source in a 14-pin butterfly package is a continuous wave, distributed feedback (CW-DFB) fiber-coupled diode laser emitting at 2.3 μm wavelength. The output power of the diode laser was 3.67 mW. The output laser was divided by wideband fiber optic coupler (FOC) into two beams with the ratio of 9:1. Ten percent of the laser power was injected into a reference cell (RC) with 3 cm optical length. After passing through the cell the laser power was detected by a photo detector (PD). A 3*f* reference channel signal was employed for locking of the laser wavelength to the peak of absorption line of CO. Using a fiber collimation package (FC) with focal length of 11 mm and a plano-convex lens (L) with a focal length of 40 mm, ninety percent of the laser power was collimated and focused between the QTF prongs at the optimal distance of 0.7 mm from QTF tip to the laser beam [14]. Micro-resonator (mR) made up by a pair of metallic tubes was used to improve the 2*f* signal [24,25]. A power meter (PM) was used to monitor the power of light passing through the QTF. A transimpedance amplifier (TA) with a resistance of 10 MΩ was used to convert the piezoelectric current to voltage. The voltage signal was used to demodulate into the 2*f* signal. The sensor system was processed by a personal computer (PC) using LabVIEW software.

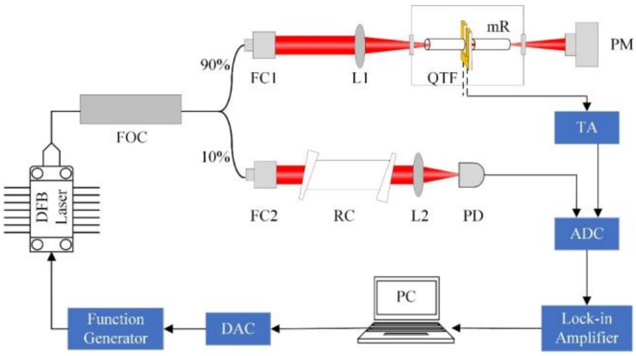

Fig. 4. Schematic of a QEPAS system for CO detection

4. Results and discussion

In the experiments, the line located at 2330.19 nm (4291.50 cm^{-1}) which is one of the strongest lines at ~2.3 μm was chosen as the target line. The amplitude of modulation was optimized and the it was found to be 0.38 cm^{-1}. Fig. 5 shows the detected 2*f* and 3*f* signals. The zero point of 3*f* siganls between two extremums was overlapping with the position of the peak of 2*f* signal at 4291.5 cm^{-1} precisely. The background noise was measured when the gas cell was filled with pure N_2. With the background noise of 1.4 μV, a signal to noise ratio (SNR) of 454.75 was obtained. The 1σ minimum detection limit (MDL) is defined as the ratio of standard gas concentration to SNR. The MDL for this CO-QEPAS sensor was 109.95 ppm (part per million) with a 1 s time constant of the lock-in amplifier. The calculated normalized noise equivalent absorption (NNEA) coefficient for the reported CO-QEPAS sensor was 1.83× 10^{-7} $cm^{-1}W/\sqrt{Hz}$。

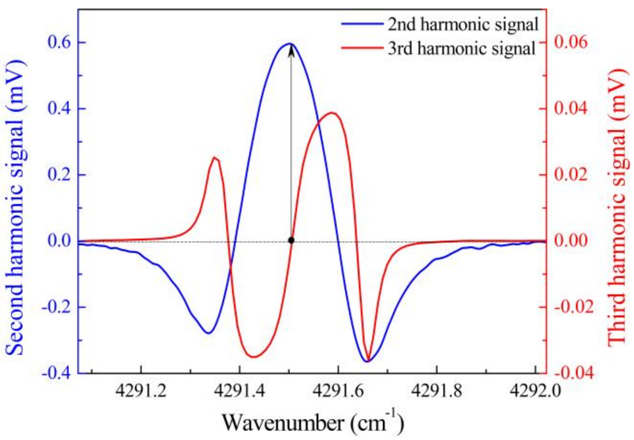

Fig. 5. Measured 2f and 3f QEPAS signals of CO

The concentration of CO was continuously monitored in a period time of 85 s. From Fig. 6 we can see that when a 5% CO: N_2 was used as analyte, the QEPAS sensor showed a stable measurement. A strong fluctuation appearing after 75 s was the result of the vibration generated in the process of changing the gas sample. When N_2 was used as the target gas, the concentration detected by sensor system was always around zero line. The system is sensitive to the change of CO concentration and has the ability to provide a real-time feedback and rapid response.

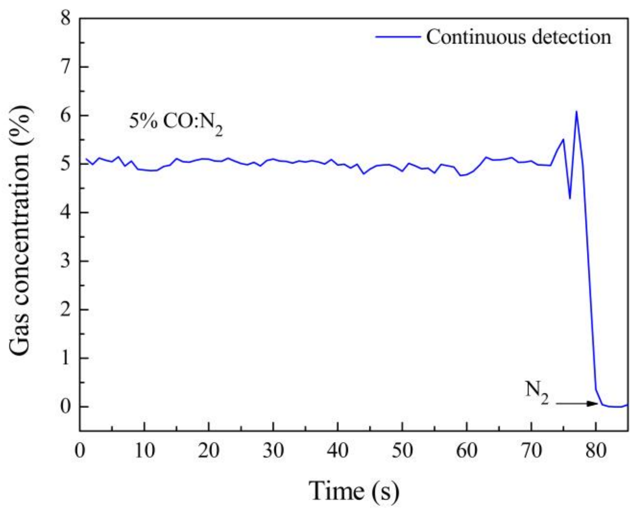

Fig. 6. A 85-second continuous monitoring of CO concentration level

5. Conclusions

In conclusion, this paper mainly introduced a continuous gas detection system of quartz-enhanced photoacoustic spectroscopy sensor based on harmonic detection technique which was controlled by LabVIEW program. By using the third harmonic signal of the reference cell, the laser wavelength was locked to the gas absorption line and the shift of the laser wavelength was well avoided. Therefore, continuous monitoring of CO concentration was performed.

Acknowledgments

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