Study of a Distributed Feedback Diode Laser Based Hygrometer Combined Herriot-Gas Cell and Waterless Optical Components

Yubin WEI^{1,2}, Jun CHANG^{1*}, Jie LIAN¹, Qiang WANG¹, and Wei WEI¹

Abstract: A distributed feedback diode laser (DFB-DL) based hygrometer combined with a long-path-length Herriot gas cell and waterless optical components was proposed and investigated. The main function of this sensor was to simultaneously improve the measurement reliability and resolution. A comparison test between a 10-cm normal transmission-type gas cell and a 3-m Herriot gas cell was carried out to demonstrate the improvement. Reliability improvement was achieved by influence suppression of water vapor inside optical components (WVOC) through combined action of the Herriot gas cell and waterless optical components. The influence of WVOC was suppressed from 726ppmv to 25ppmv using the Herriot gas cell. Moreover, combined with waterless optical components, the influence of WVOC was further suppressed to no more than 4ppmv. Resolution improvement from 11.7 ppmv to 0.32 ppmv was achieved mainly due to the application of the long-path-length Herriot gas cell. The results show that the proposed sensor has a good performance and considerable potential application in gas sensing, especially when probed gas possibly permeates into optical components.

Keywords: Herriot gas cell; hygrometer; wavelength modulation spectroscopy; waterless optical components; water vapor inside optical components

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1. Introduction

Reliable and high-resolution measurement of gases of interest becomes more and more important in the field of scientific research and facility manufacture. Particularly, trace water vapor qualification analysis is indispensable in a wide of applications, ranging from polymer fabrication [1], environment analysis [2] to chemical production [3]. Because of remote detection capability, safety in

hazardous environments, and immunity to electromagnetic radiation, the distributed feedback diode laser (DFB-DL) based hygrometer attracted the widest applications. In the recent years, many techniques were developed to improve the measurement reliability and resolution. For example, the utilization of differential value of two adjacent absorption peaks solved the difficulty of choosing reference point [4]. The utilization of combination of

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wavelength scanning and intensity modulation significantly improved the detection resolution [5]. The application of wavelength modulation spectroscopy (WMS) could achieve high resolution detection of trace gases at atmospheric pressure [6]. The utilization of the improved WMS technique achieved rapid measurement of water vapor at high temperature [7].

Almost all of DFB-DL based hygrometers consist of several optical components, such as light source (DFB-DL), fiber collimators, photoelectric detector. However, it was found in [8] that water vapor in these optical components seriously influenced the measurement result not only in resolution but also in reliability. To realize reliable and high-resolution measurement of water vapor, an approach, namely the photo detector (PD) matching, had been reported to achieve preliminary suppression for the influence of water vapor inside optical components (WVOC). However, it is found in practical application the suppression effectiveness of the above approach depends on the match level between the two PDs, which is tedious and not guaranteed.

In this paper, an alternative approach consisting of Herriot cell and waterless optical components is investigated. It can observably improve the reliability and resolution simultaneously. The influence of WVOC can be diluted by introducing a Herriot cell with an effective length of 3 meters. Here we describe the experimental system and the mathematical justification of this approach and demonstrate the validity through exhibiting the improved measurements made by this approach.

2. Experimental details

Figure 1 shows the experimental system. A DFB-DL (with a linewidth of 3 MHz) is used as the light source. The inside temperature is controlled by a chip LTC 1923. The driving current consists of a 20-Hz sawtooth waveform and a 2-kHz sinusoidal waveform, and it is produced by a home-built signal

generator using a chip LPC 1758. The sawtooth waveform ranges from 23 mA to 78 mA. As a result, a sweep range of about 280pm is given to scan the absorption line of water vapor at 7306.75 cm⁻¹. At the same time, the sinusoidal current is used to accomplish wavelength modulation of the DFB-DL. The output of the DFB-DL propagates through a gas cell to interact with water vapor molecules. To introduce a long optical absorption path, a multiple-pass Herriot cell is used. The 34 traversals inside the Herriot cell give a path length of 3 meters. The long optical path length not only improves the effective absorbance during the interaction, which is beneficial to improve the measurement resolution but also dilute the influence of WVOC, which is very important to improve the measurement reliability. After the interaction, the laser beam is detected by an InGaAs PD whose output is fed to RS850 digital lock-in amplifier (LIA). To implement the WMS technique, the second harmonic detection is performed with the LIA. To avoid variation of the phase difference between the WMS derivative signal and reference signal, the reference signal is synchronously generated by the same home-built signal generator. The final output is observed and captured by a computer.

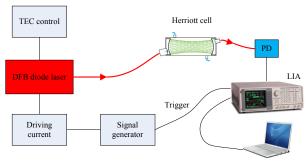


Fig. 1 Experimental setup.

3. Analysis of reliability and resolution improvement

The description of laser beam attenuation due to wavelength dependent absorption for a gas absorption line is given by Beer-Lambert's law [9], $I_t(v)=I_0(v)\exp[-\alpha(v)CL]$, where $I_0(v)$ and $I_t(v)$ are the

216 Photonic Sensors

incident and transmitted laser beam intensities, respectively, $\alpha(v)$ is the wavelength independent absorption coefficient, C is the mole fraction concentration, and L is the optical path length. For WMS, the simultaneous frequency and amplitude of the modulated laser beam can be represented by

$$v(t) = v_c + \Delta v \cos(\omega t) \tag{1}$$

$$I_0(t) = I_{0c} + i_0 \cos(\omega t + \psi)$$
 (2)

where v(t) is the instantaneous optical frequency, $I_0(t)$ is the laser emission intensity, and ψ is the phase separation [10] between the intensity and optical frequency modulation. v_c and I_{0c} donate the slowly-varying central frequency and intensity, as the laser scans across the absorption features. ω is the sinusoidal modulation frequency, Δv and i_0 are the maximum small-amplitude excursions of the v(t) and $I_0(t)$ around v_c and I_{0c} .

In the implementation of the WMS technique, the signals at the output can be found using Fourier cosine series expansion of the absorbance line shape function, $\alpha(v)CL$ [11]. The result for the primary second harmonic term is shown as

$$I_{2\omega}(v_c) = \frac{i_0}{2} H_3(v_c, \Delta v) \cos(2\psi + \varphi) + I_{0c}H_2(v_c, \Delta v) \cos(\psi + \varphi) + \frac{i_0}{2} H_1(v_c, \Delta v) \cos\varphi$$
(3)

where H_1 , H_2 , and H_3 denote three Fourier components obtained for the absorbance line shape function. φ designates the phase difference between the detection signal and the laser emission intensity. Although the components of H_3 and H_1 on the measurement axis of H_2 can distort the detected profile of the detected second harmonic term [12], the amplitude of the detected second harmonic signal at the line center is immune to the components of H_3 and H_1 . This is because the amplitudes of H_3 and H_1 are zero at the absorption peak [11]. Thus the amplitude of the detected second harmonic (i.e. the value of the detected second harmonic signal at the absorption peak) can be expressed as

$$I_{2\omega}(v_p) = I_{0p} H_2(v_p, \Delta v) \cos(\psi + \varphi)$$
 (4)

where v_p and I_{0p} donate the frequency and intensity at the absorption peak. To simplify the expression of the relationship between the detected second harmonic amplitude and absorbance, (4) can be rewritten as

$$I_{2\omega}(v_p) = \kappa \alpha(v_p) CL. \tag{5}$$

The above expression is applicable only when the absorbance is not too high. For the detection of water vapor at $7306.75 \, \mathrm{cm}^{-1}$ in this research, the above expression is applicable when C is no more than 2000 ppmv. κ in (5) designates the conversation coefficient, which is relative to the beam intensity at the absorption peak, values of ψ and φ , and Fourier component H_2 .

According to the analysis in [8], the water vapor inside the optical components can influence the measurement result. Combined with (5) in the WMS technique, the influenced expression of the water vapor concentration should be

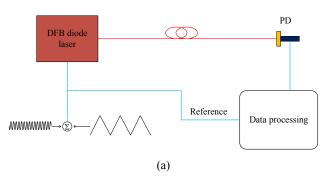
$$C = \frac{I_{2\omega}(v_p)}{\kappa\alpha(v_p)L} + \frac{L_{\text{eq}}}{L} \times C_{\text{eq}}$$
 (6)

where L_{eq} and C_{eq} donate the equivalent absorption length and concentration of the water vapor inside the optical components. An extended optical absorption length can simultaneously improve the measurement reliability and resolution. Firstly, during the practical application, the detected harmonic signal is directly used to invert the wanted water vapor concentration. However, system noises (optical noise and electrical noise) are accumulated and added in the detected harmonic signal. Generally speaking, the system noises have nothing to do with the absorption length. According to (6), for the same optical gas sensor, the measurement resolution can be significantly improved using a longer optical absorption length. Secondly, the influence of the water vapor inside the optical components is relatively certain and invariable. Similarly, the measurement reliability can be improved by weakening the influence of the water vapor inside the optical components.

4. Experimental results

4.1 Existence of WVOC

To demonstrate the severity of existence of the water vapor inside the optical components, a simple test was organized. The simple schematic of the test is shown in Fig. 2(a). Laser light from DFB-LD directly couples on the PD. The most distinctive characteristic is there is no gas cell in the test. The process of the detected signal is the same with the introduction above.



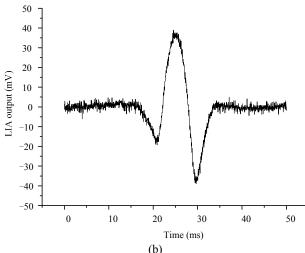


Fig. 2 Test of existence of the water vapor inside the optical components: (a) test setup and (b) measured second harmonic signal without the gas cell.

Based on the traditional understanding, there should be no absorption during this setup. However, the detected result shown in Fig.2 (b) subverts our preliminary cognition. Not only the existence of WVOC badly impacts the measurement result during the practical application of an optical fiber

hygrometer, but also variation of the inside water vapor can further impact the measurement.

4.2 Reliability and resolution improvement

To fully exhibit the influence of the water vapor inside the optical components especially the improvement of reliability resolution, a and comparison research was demonstrated experimentally between the normal transmissiontype gas cell with a 10-cm effective path length [8] and Herriot gas cell with a 3-m effective path length. Figure 3(a) shows the structure of the normal gas cell comprising two commercial fiber collimators used in communication. One collimator is used to transfer laser beam into parallel light inside the gas cell, and the parallel light interacts with the probed gas molecules and possesses information of characteristics associated with the probed gas molecules. The other one is used to receive the signal light and couple it into the fiber. The two fiber collimators are fixed on a stainless steel tube to guarantee reliable alignment and achieve low coupling loss (less than 0.5 dB). Holes of the stainless steel tube are used the realize gas exchange.

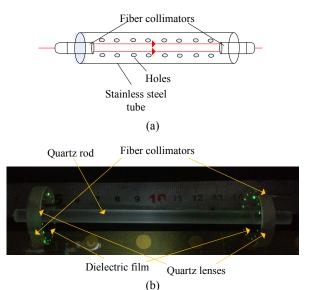


Fig. 3 Schematic of gas cell structure: (a) normal transmission-type gas cell with an effective path length of 10 cm and (b) Herriot gas cell with an effective path length of 3 m, the two quartz lenses are coated with the dielectric film.

218 Photonic Sensors

Figure 3(b) shows the structure of the Herriot cell. Two quartz lenses with a diameter of 25.4 mm are fixed with a straight column quartz rod. The two quartz lenses coated with the dielectric film have the same curvature radius of 100 mm and are separated with a distance of about 90 mm. A fiber collimator with a diameter of 1.3 mm is used to introduce light into the Herriot gas cell. After adjusting the incidence angle, 17 reflections exist for each lens. Another identical fiber collimator is used to collect the signal light and couple it into fiber. As a result, the effective optical path length has been extended to about 3 meters, and the insertion loss is lower than 1 dB.

During the comparison research. measurement reliability and resolution were studied. The reliability improvement reflected in influence suppression of WVOC. A linearity test was arranged. A water vapor generator (DG-70, Shanghai Hema, China) was used to generate sample gases with different water vapor concentrations ranging from 20 ppmv to 430 ppmv. To change the inside water vapor concentration, sample gases were introduced into the gas cell at a flow rate of 100 mL/min at 1 atm and (296 ± 0.5) K. The overflowing sample gas from gas cell was used to monitor the water a vapor concentration bv moisture (S8000, MICHELL, UK). Precision of S8000 is within 1 ppmv in the range of 1 ppmv -2000 ppmv.

The measurement result is shown in Fig. 4. Amplitude (in mV) is proportional to the water vapor concentration. The solid line named "normal transmission-type gas cell" is the linearity test result using the 10-cm normal transmission-type gas cell, and the dot line named "Herriot gas cell" is the linearity test result using the 3-m Herriot gas cell. It can be seen from Fig. 4 that both the lines do not pass through the origin of coordinates (0, 0), and this is caused by the influence of WVOC. That is to say influence of WVOC can be reflected by the intercepts of the measured linearity lines. After the

linear fitting process slope of solid line is calculated to be 0.11 mV/ppmv, thus the intercept of solid line is 79.9 mV corresponding to a water vapor concentration of 726 ppmv in a 10-cm path length. While the slope of dot line is calculated to be 3.38 mV/ppmv, and the intercept of solid line is 85.1 mV corresponding to a water vapor concentration of 25 ppmv in a 3-m path length. Apparently, the influence of WVOC on the measured probed water vapor concentration has been significantly suppressed.

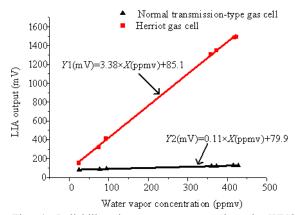
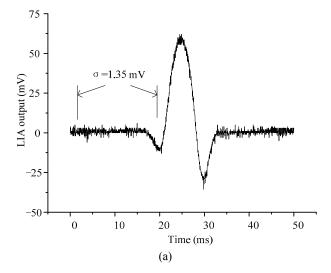


Fig. 4 Reliability improvement test through WVOC suppression (the lower line is for a 10-cm transmission-type gas cell, and the upper line is for a 3-m Herriot gas cell).

As for the resolution improvement test, the resolution [with signal to noise ratio (SNR) =1] is obtained through deducing the SNR. Given the existence of WVOC, the deducing process is not based on a certain water vapor concentration. On the contrary, the signal amplitude in SNR calculation is derived from two different amplitudes (in Fig. 4) corresponding to two water vapor concentrations, which are measured by S8000. The noise amplitude is derived from the WMS profile corresponding to one of the two water vapor concentrations shown in Fig. 5.

From the above test and process, the resolution of measurement using the normal transmission-type gas cell is calculated to be 11.7 ppmv, and that of measurement using the Herriot gas cell is 0.32 ppmv. Thus reliability can obtain significant improvement using a long path length gas cell.



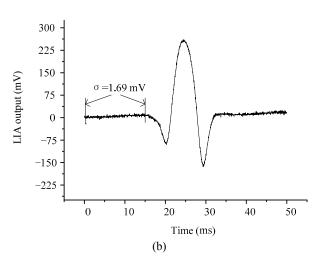
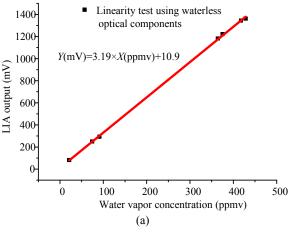


Fig. 5 Detected WMS profiles: (a) corresponds to the normal transmission-type gas cell with a water concentration of 94 ppmv and (b) corresponds to the Herriot gas cell with a water concentration of 92 ppmv.

4.3 Further improvement using waterless optical components

Although a long optical path length gas cell can apparently suppress the influence of WVOC, there still exists an apparent influence, which is shown in Fig. 4. Thus waterless optical components were designed and adopted to further suppress influence of WVOC. A 14-pin butterfly packaged DFB-DL and a PD with the same package were used. The big difference between the two waterless optical components and normal ones is that the inside space is filled of high purity nitrogen. As a result, inside water vapor content could be rather low.

To obtain the efficiency of water vapor suppression using these waterless optical components, a test was carried out, and the test result is shown in Fig. 6 (a). It is apparent that the intercept of the linearity test has been suppressed to 10.9 mV corresponding to no more than 4 ppmv. Figure 6(b) shows the detected LIA output without the gas cell and a result of fitting procedure based on the Fourier analysis of the Lorentzian profile.



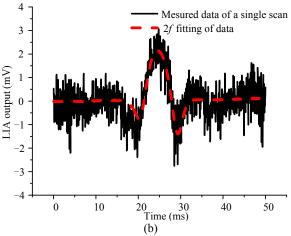


Fig. 6 Further suppression results for influence of WVOC using waterless optical components: (a) linearity test and (b) situation of no gas cell.

5. Conclusions

We proposed and demonstrated a technique comprising Herriot Cell waterless optical components. Reliability and resolution of the DFB-DL based hygrometer can be simultaneously improved by this technique. A comparison test between a 10-cm normal transmission-type gas cell

220 Photonic Sensors

and this 3-m Herriot gas cell was carried out. As a result, the influence of WVOC had been suppressed from 726 ppmv to 25 ppmv. Moreover, waterless optical components were used to further suppress the influence of WVOC, and the influence had been further suppressed to be no more than 4 ppmv. In addition, the measurement resolution had been improved from 11.7 ppmv to 0.32 ppmv.

In summary, the reliability improvement had been achieved through suppressing the influence of WVOC using a long path length Herriot gas cell and waterless optical components. The resolution improvement had been achieved through enhancing absorbance using a long path length Herriot gas cell. The proposed sensor has potential applications in the design of the reliable and high-resolution gas sensor, especially when permeation of probed gas into optical components exists.

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