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Trace Humidity Sensor based on Quartz-Enhanced Photoacoustic Spectroscopy

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Abstract: A compact trace humidity sensor based on quartz enhanced photoacoustic spectroscopy (QEPAS) technique was designed and characterized. A sensitivity of $2.5 \times 10^{-9} \text{ cm}^{-1} \text{ W/Hz}^{1/2}$ was achieved. An influence of oxygen presence on the detected signal was observed.

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1. Introduction

A number of industrial applications including semiconductor manufacturing and natural gas transportation require gases with water content at or below a few parts per million by volume (ppm) level. Therefore, there is a need in technology for trace water vapor quantification that can be implemented into a portable sensor suitable for use in an industrial environment. Quartz enhanced photoacoustic spectroscopy (QEPAS) was recently applied to detect trace concentrations of several gases using both near-infrared and mid-infrared spectroscopic sources [1, 2]. QEPAS based gas sensors can analyze extremely small gas samples, they are compact and immune to environmental acoustic noise, as well as to the spectroscopic source power fluctuations and unintentional interference occurrences in the optical system. These features potentially allow development of portable and even hand-held trace gas sensors, provided that suitable laser sources are available. In this work the performance of a compact trace humidity sensor based on QEPAS approach in the near-infrared (overtone molecular absorption) region is presented.

2. Sensor configuration

The trace humidity sensor architecture was based on a concept outlined in [3], and its optical layout is shown in Fig. 1. Presence of water vapor in a gas sample was detected via sound generated in the sample when the wavelength-modulated laser radiation was absorbed. A fiber-coupled DFB laser in a 14-pin butterfly package operating at $\lambda = 1.37 \text{ }\mu\text{m}$ (NEL model NLK1E5E1AA) was used as the excitation source. The laser diode temperature and current were selected so as to target an H_2O absorption line at 7306.75 cm^{-1} . The absorption detection module (ADM) consisted of a quartz tuning fork (TF) acting as a piezoelectric transducer with a resonant frequency $f_0 \approx 32.7 \text{ kHz}$ and a pair of glass tubes with a combined length of $\sim 5 \text{ mm}$ which formed an acoustic resonator at the same frequency. A 99:1 fiber beamsplitter was used to direct main part of the laser radiation to the ADM while $\sim 1\%$ was sent to a reference channel for wavelength stabilization of the diode laser. The reference cell was 5 cm long and filled with a sample of room air (50% humidity) at 100 Torr. This cell was used for line locking when reduced pressure measurements were performed (50 – 200 Torr). When the measurements were performed at close to atmospheric pressure (500-800 Torr), the reference cell was removed and an ambient air in the gap between the fiber collimator and the photodiode served as a spectral reference. Due to air conditioning in the laboratory, summer time humidity of ambient air is stable at a 50% level. Radiation power delivered to the ADM was $\sim 9.5 \text{ mW}$. A fiber collimator and $f = 36 \text{ mm}$ lens were used to focus the beam between the TF prongs. A spherical mirror ($R = 50 \text{ mm}$) was installed after the ADM to back-reflect the laser radiation and thus enhance the photoacoustic signal. Laser current control and data acquisition were performed by an autonomous electronics module with the capability to communicate with a PC by means of a RS232 serial interface [3]. In addition, a lock-in amplifier (SRS model SR830) and a function generator (SRS model DS345) were employed for the initial sensor setup and calibration. Nitrogen gas with a specified humidity of 44.2 ppm (Scott Specialty Gases) was used as a calibration standard.

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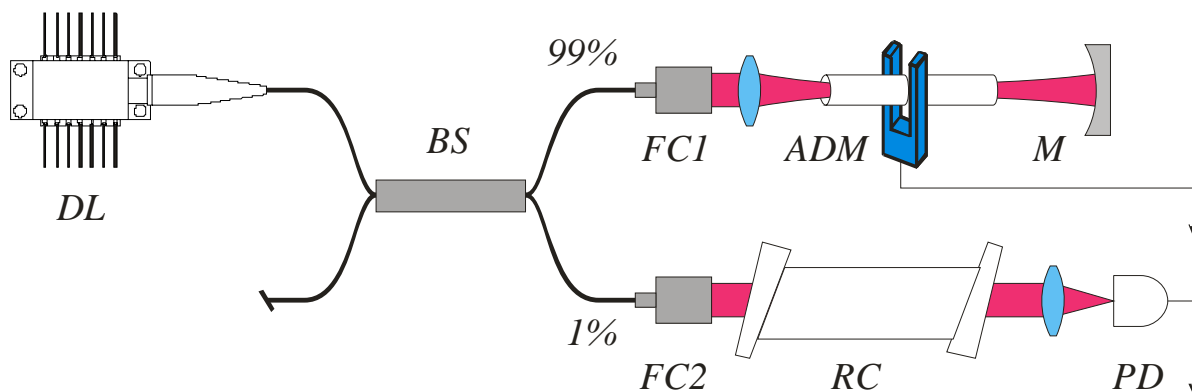


Fig. 1. The QEPAS-based trace humidity sensor architecture. DL – fiber-coupled diode laser, BS- fiber beamsplitter, FC1,2 – fiber collimators, ADM – absorption detection module, M – back-reflecting spherical mirror, RC – reference cell, PD – photodiode.

3. Results and discussion

QEPAS signal was measured at different pressures of the calibration gas to determine the gas pressure and laser current modulation depth for the highest signal to noise ratio (SNR). It was found that the best conditions correspond to a pressure of 60 Torr, which indicates fast V-T relaxation of a vibrationally excited H_2O molecule [3]. An example of the QEPAS $2f$ wavelength modulation spectrum acquired near 7306.75 cm^{-1} H_2O absorption line is shown in Fig. 2a. Baseline noise in this spectrum is too low for accurate detection limit assessment, and is influenced by the limited dynamic range of the lock-in amplifier. Therefore, a separate scan was performed for the far wing spectral region of this absorption line with 100 times lower lock-in amplifier range settings (Fig. 2b).

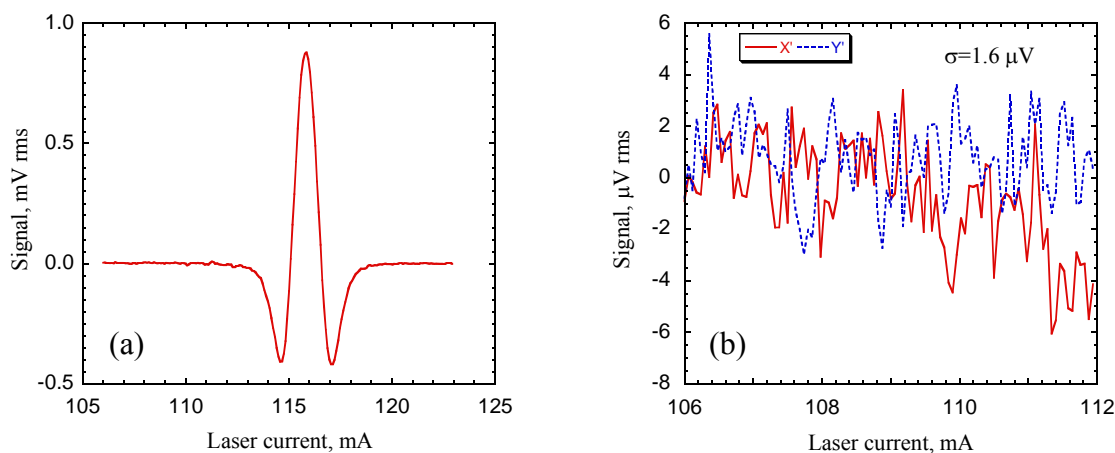


Fig. 2. QEPAS signal detected for 44.2 ppm of H_2O in N_2 at 60 Torr pressure (a). A wing of the same absorption line, data acquired at the same conditions but higher lock-in amplifier gain (b). X' and Y' are correspondingly in-phase and quadrature signal components. Lock-in amplifier time constant $\tau=1\text{ s}$.

Based on these data, the $\text{SNR}=550$ and noise-equivalent H_2O concentration for these conditions is 90 ppb (parts per billion by volume). The normalized noise-equivalent absorption coefficient is found to be $\text{NNEA}=2.5\times 10^{-9}\text{ cm}^{-1}\text{ W}/\text{Hz}^{1/2}$. This number matches sensitivity levels achieved in conventional photoacoustic spectroscopy. While 60 Torr gas pressure results in the highest sensitivity, the sensor can also operate at normal atmospheric pressure. NNEA measured at 800 Torr is $5.5\times 10^{-9}\text{ cm}^{-1}\text{ W}/\text{Hz}^{1/2}$.

It is known that a gas carrier composition can influence V-T relaxation rate of the trace species and thus change the photoacoustic response. We compared QEPAS signals for the same H_2O concentrations in pure N_2 and in air. It was found that the sensor response to H_2O in air is 2.7 times lower compared to pure N_2 . This change is most likely due to the presence of O_2 and a resonant energy transfer channel from vibrationally excited H_2O to O_2 . Relaxation of

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the vibrational excitation of oxygen molecule is very slow, thus the energy transferred to O₂ does not input into the photoacoustic signal. A similar effect was earlier reported for photoacoustic detection of CO₂ in nitrogen [4].

4. References

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