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Intracavity gas detection with fiber loop ring down spectroscopy

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ABSTRACT

In this study, an easy to construct and sensitive intracavity fiber loop ring down spectrometer was built by commercially available basic components and its application on chemical sensing is presented. System performance was investigated by using various acetylene/nitrogen gas mixtures as a function of total pressure (between 25 and 65 psia) in sensing region and a calibration curve is established. The instrument can achieve a limit of detection of 0.1% without a FBG and/or EDFA or a need for a complicated design. Moreover, the detection limit can be further improved by working at higher pressures. With the current design of the system, the change in type and/or concentration of species in the sensor region can be monitored in real time.

1. Introduction

Optical fiber based sensors attract great interest due to their desirable features such as being fast, compact, sensitive and reliable in addition to having high resolution and low detection limit. Such an interest fuels development of highly anticipated, multi-functional optical fiber based measurement technique called fiber loop ring-down (FLRD) spectroscopy for uses in diverse application areas [1,2]. FLRD is a derivative of cavity ring-down technique and is using an optical fiber as a waveguide. It is a time domain spectroscopy technique in which a laser pulse is trapped in a fiber cavity with low losses enabling multiple interaction with the sample. FLRD technique finds applications in sensing of strain [3], refractive index [4], temperature [5], pressure [6], and most importantly in sensing of chemical [7] and biological species [8–10].

FLRD technique monitors decay of a laser pulse in the cavity due to inherent system component losses and absorption of the sample in sensor region as trapped laser pulse makes multiple rounds in the loop. This decay in general is monitored by ring down time (RDT) which is a specific duration corresponding to the time that passes for the incident laser pulse power to decrease to 1/e of its initial level. RDT depends on intrinsic fiber loss, coupler losses, splice losses, and characteristics of sensor region. One of the best advantages of FLRD technique is its *insensitivity to laser power fluctuations and detector response* [8]. Multiple passes of a pulse that is trapped in the cavity in an FLRD system increases interaction path length and secures stability of measurement result. Signals that are measured by a photodetector at each round carry the same information about sensor region thus FLRD promises a high sensitivity.

With its great potential FLRD has already found applications in numerous areas with its aforementioned unique properties. One of the first applications of FLRD was in telecommunication, where optical loss is a very important issue and must be low for long distance information transmission. Here, loss measurement less than 0.1% is quite difficult with usual techniques and, with its multi-pass nature FLRD was used to measure optical loss due to coupler, optical fiber, and fiber splice with great accuracy [11]. Another area where FLRD finds application is in monitoring of global warming gases. Global warming gases such as CO_2, CH_4 and CO are detectable with gas chromatography but remote detection is not yet possible, thus, FLRD spectrometer has potential to sense global warming gases remotely [12]. FLRD recently finds application in detection of chemicals and their concentrations as lasers become available in different colors suitable for the chemical identification. A commonly used chemical during instrument development is acetylene for testing spectrometer's stability and sensitivity at around 1534 nm. Zhang et al. used erbium doped fiber amplifier (EDFA) and fiber Bragg grating (FBG) in their FLRD spectrometer set-up to increase the sensitivity and were able to detect 1% acetylene with a 1 cm long sampling cell [13]. With a 3 km optical fiber and 25 cm gas cell FLRD setup, Zhao et al. were able detect 0.1% acetylene with 1.02% stability and 0.18% single point reproduciblity [14]. Andrews et al. also used EDFA in addition to band pass filter on a nested loop in their FLRD setup for detection of acetylene in helium at 1532 nm using phase shift method and could quantify 50 ppm acetylene in helium [15]. Similarly, with an introduction of EDFA and FBG to their setup with a 65 mm long gas cell Zhao et al. were able to detect 0.1% acetylene in N_2 with a deviation of less than

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0.4% [16]. However, there is still much room for improvement both in sensitivity and in simplification of the FLRD set-ups toward development for commercial use of the instrument. For instance, the effect of sample gas pressure in the sensing region on the system sensitivity have not been studied before which may have important implications for designing FLRD systems with pre- or on site-concentration capabilities.

In this study, we introduce a sensitive intracavity based FLRD spectrometer with high reproducibility and low deviation in RDT and present its application on quantitative detection of acetylene. The spectrometer has a simpler configuration and is constructed from commercial low cost components. In addition, sensing region of the instrument was designed to allow high pressures, which makes it possible to increase the concentration of the analyte by increasing the amount/pressure of the sample gas. The following sections describe FLRD spectrometer's sensor region, data collection procedure, data processing, and instrument stability along with its application on acetylene detection.

2. Experimental

Nitrogen (N_2, 99.99%), acetylene (C_2H_2, 98%) and 0.1%, 1%, 5%, and 10% (v/v) C_2H_2 in N_2 gas mixtures were purchased from Linde Inc. and used as is without any further purification. The mixture gases have 2% tolerance in C_2H_2 amount. Our FLRD set-up is constructed with a 4 ns pulsed, 1534 nm (6 nm FWHM) laser (Cobolt Tango) operating at 3 kHz, a fiber coupled lens, 25 m single mode fiber, 99:1 coupler, isolator, a sensor region (0.718 ± 0.002 dB calculated loss) created by two collimators (Thorlabs 50–1550 A), two photodiodes (EOT 3010, DET08CFC), and an oscilloscope (Tektronix MSO 4104). Fig. S1 in supporting information presents the laser spectrum and its overlap with C_2H_2 absorption lines. The details of the components used in the set-up are also given in supporting information.

The spectrometer configuration and the sensor region details are given in Chart 1. In the FLRD set-up, the laser pulse from free space is guided to fiber via fiber-coupled lens. One percent of the laser pulse is coupled to the cavity with the coupler and guided to the sensor region. The rest of incident laser power is directed to a photodiode and used as trigger for the oscilloscope. After the pulse passes through the sensor region, 1% of the light travelling in the cavity is directed to a second photodiode by an isolator for recording the signal level at each round trip. The rest is directed to the loop for multiple pass through the sample region until all the light in the loop decays. The secondary purpose of the isolator is to prevent the back reflection off of the collimator reaching to the photodiode.

The spectrometer monitors decay of a laser pulse in the cavity due to inherent system component losses with and without sample absorptions as the trapped laser pulse makes multiple rounds in the loop. RDT depends on intrinsic fiber loss, coupler loss, splice losses, and absorption by a sample. With its multi pass nature in the cavity, interaction path length is increased enabling stable measurements at low absorptions by the sample. Inset in Fig. 1a presents a representative data of the pulse intensity after each round, forming a pulse train. Here the signal level of each pulse follows an exponential decay as expected due to the inherent losses of the instrument components at each round. The ring down time and corresponding loss of the closed loop, without a sensor region, are 1046.3 ± 0.8 ns and 0.5977 ± 0.0004 dB, respectively, (see Supporting Information Fig. S2 for decay train of incidence pulse through the closed loop).

The photodetector transfers signal levels to the oscilloscope and data is recorded with help of a Matlab code. Each data set is collected with 512 averages at a 50 Ω terminated input with 50 mV voltage scale in the oscilloscope. A study on set scale of the oscilloscope to the dynamic range and its effect on the signal-to-noise (S/N) showed that 50 mV scale has the highest S/N in calculated RDTs with the lowest standard deviation. Once the data for the ring down time is collected the background response of the detector is identified as in red trace in Fig. 1a, which is due to the detectors inherent response. Even though the detector response did not have a significant contribution to the signal level recorded as seen in the actual data given in the inset, it is corrected as follows. The data corresponding to the detectors response is separated and fitted to an exponential.(Fig. 1b) The experimental data (now on called data) is then corrected by subtracting the fit from the collected one.(Fig. 1c) Next, the peak amplitude of each pulse in the data is determined. The first 9 peaks were discarded since they were out of scale in the 50 mV voltage scale of the oscilloscope. Finally, the peak points are fitted to Eq. (1) to extract the RDTs.(Fig. 1d)

$$I = I_0 * exp^{-t/\tau} \quad (1)$$

Here I_0 and I are initial amplitude and amplitude at time t, and τ corresponds to the ring down time. The data presented in Fig. 1 is for N_2 with a ring down time of 485.4 ± 0.6 ns or 1.3152 ± 0.0016 dB loss per round trip. One of the main factors determining the ring down time (and sensitivity in part) of the spectrometer is the path length of the sensor region. The loss increases significantly when the distance between the collimators is beyond a certain length due to diverging/scattering light after the first one. Thus, the optimized distance is found to be 9 mm for the highest sensitivity while keeping the loss as low as possible. Regardless, the two collimators add extra loss compare to the closed loop like pair insertion and insertion losses, which is less than 0.33 dB at 9 mm separation. The sensor region is sealed with a plexiglass housing to which collimators are fixed by epoxy glue and gas inlet and outlet ports are fixed by high pressures fittings. The decay train is monitored and the collimators are constantly aligned during curing stage of the epoxy. Once dried, the sampling region is checked for gas leakage.

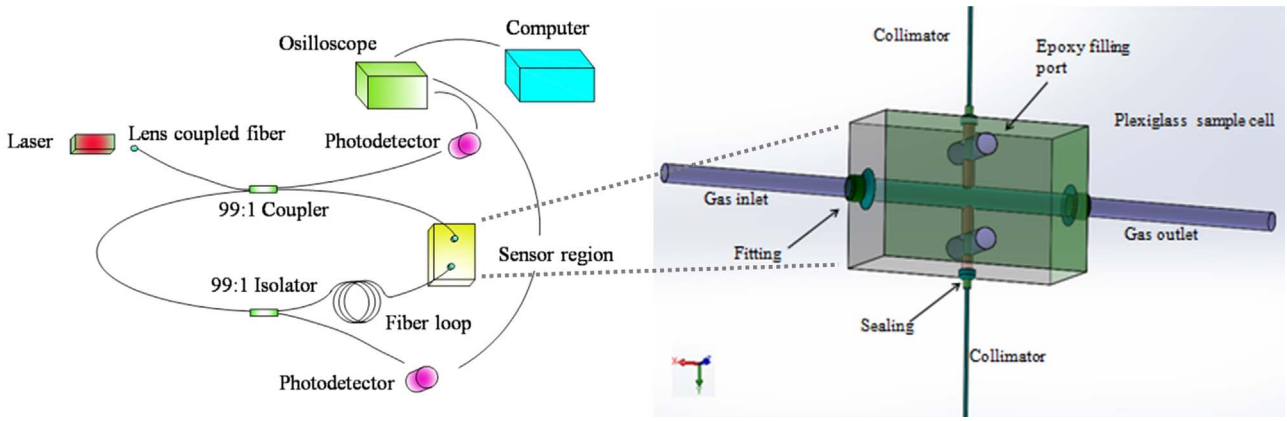

Chart 1. Schematic configuration of the intracavity FLRD spectrometer (left) and the sensor region (right).