Gas sensing demonstration by using silica high-mesa waveguide with amplified cavity ring down spectroscopy technique

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Abstract: For realizing compact breath sensing device, we have proposed a silica high-mesa waveguide for the gas-cell (breath detection part) because of its low propagation loss. It is, however, still difficult to make breath-sensing due to its total insertion loss because the required length of the waveguide reaches very long of approximately several 10 cm–1 m for the small portion of gas, and thus gas-sensing has not been achieved so far. To compensate the insertion loss, we utilize “amplified” CRDS (cavity ring down spectroscopy) technique to realize gas-sensing in this paper. As a result, we could successfully confirm the gas-sensing of CO\(_2\) with using the waveguide gas-cell for the first time.

Keywords: silica high-mesa waveguide, infrared absorption, gas sensing device, amplified CRDS

Classification: Optoelectronics, Lasers and quantum electronics, Ultrafast optics, Silicon photonics, Planar lightwave circuits

References

1 Introduction

Easy-to-use health-check equipment has been desired for daily healthcare recently, especially due to the aging generation issue. We focus on breath content as a disease marker, because the breath contains various diagnostic information [1, 2, 3, 4], such as for digestive disease [2], lipid peroxidation [4], and lung disease, particularly asthma [2, 3], with less stress to human beings. When downsizing such a breath-sensing system, however, a gas-cell becomes a major bottleneck since it consists of a long optical path length (approximately 1 meter long) for ppm-order disease-marker volatile gases in the breath [2, 3, 4]. For instance, the required length reaches 1 m over in case of detecting 10 ppm CH₄ (disease marker of the digestive disease [2]) in the breath. In order to solve this problem, we have proposed a silica high-mesa waveguide [5] for the gas-cell (breath detection part). The gas-sensing utilizing waveguide have been studied by many research institutes [6, 7, 8, 9, 10, 11]. Similarly, in measuring method exploiting waveguide, a wide variety of method is also studied such as infrared absorption [6, 7, 8], Raman scattering [9], surface plasmon [10], and polymer colorimetric analysis [11].
Among them, infrared absorption spectroscopy is attractive in the capability of real-time measurement and the superior ability to distinguish structurally similar gas molecules (ex. CO$_2$, CO, and others). And high-mesa waveguide structures have outstanding features for the breath sensing. For instance, high-mesa have a certain portion of the optical field profiles out of the waveguide [12, 13, 14], a very small curvature-radius [5, 13], easiness of fabrication and easy-to-contact to sample gas. Thus, we have proposed a silica high-mesa waveguide for the gas-cell (breath detection part) because of its low propagation loss. The implemented waveguide showed extremely low propagation loss of 0.02 dB/cm with portion of the propagation light out of the waveguide $\Gamma_{\text{out}}$ (approximately 2.2%) [15]. It is, however, still difficult to sense breath content due to its total insertion loss because the required length of the waveguide reaches very long of approximately several 10 cm–1 m for the small portion of gas, and thus gas-sensing has not been reported so far. To compensate the insertion loss, we utilize “amplified” CRDS (cavity ring down spectroscopy) technique in this work [16]. As a result, we could successfully confirm the gas-sensing of CO$_2$ with using the waveguide gas-cell for the first time.

### 2 Silica high-mesa waveguide

Fig. 1 shows the waveguide cross-sectional view (a) and the optical field profile (b) of the silica high-mesa waveguide. As is shown in the figure, it is consisted from GeO$_2$-doped SiO$_2$ core with SiO$_2$ cladding. Relative refractive index difference $\Delta n$ of the waveguide in the perpendicular direction is set to be 2.5%. Low index contrast between air and the core may contribute to less suffering from the side-wall roughness, in addition to the material intrinsic low absorption loss. The lateral confinement of the waveguide is realized by so-called high-mesa structure. As the side-wall of the core directly faces to the outside, a certain portion of the optical field profiles out of the waveguide (here we define this value as $\Gamma_{\text{out}}$), which is utilized for gas absorption as is shown in Fig. 1(b) although $\Delta n$ in the perpendicular direction is not so high.

For the actual implementation, layer structure was formed by flame hydrolysis deposition and then regular reactive ion etching technique [17] was used to form “high-mesa”. The etching depth was approximately 13.5 µm. The implemented waveguide showed extremely low propagation loss of 0.02 dB/cm and $\Gamma_{\text{out}}$ of 2.2%.
at 1572 nm [15]. To make sure, this wavelength corresponds to one of the peak-wavelengths of CO₂ (major content of human breath) infrared absorption. This low propagation loss is realized due to its low refractive index contrast in the lateral direction.

3 Gas sensing using amplified CRDS

As is explained in chapter 1, we exploit amplified CRDS (cavity ring down spectroscopy) [18, 19, 20]. Conventional CRDS obtains ring-down pulse by injecting optical pulse into gas cavity of which both ends are consisted from high reflection mirrors as shown in Fig. 2. As reflected back light corresponds to the extension of effective optical path length, this method is useful for highly sensitive detection. During the reflection happens, a small portion of the optical pulse comes out from the high reflection mirror and is detected as output pulses. In such a manner, so called ring-down pulse intensity follows exponentially decreasing curve shown in right-hand side of Fig. 2. In case a certain portion of detecting gas exits in the cavity, the ring-down time, which is defined as a decay time of 1/e pulse intensity compared to the initial pulse one, decreases due to gas absorption (Fig. 2(b)) compared to the case of vacuum as shown in Fig. 2(a). The gas concentration is estimated by the ring-down time difference between the ring-down time without gas absorption and with gas absorption [21].

However, it is required long optical path length to obtain sufficient ring-down time difference (that correspond more than pulse round-trip time inside the cavity) because gas absorption is very low such as 0.0004 dB/cm at 10 ppm of CH₄ which is one disease-marker in breath. For instance, to measure 10 ppm of CH₄ requires over 500 cm of optical path length in CRDS. Then, gas absorption corresponds to 0.2 dB absorption loss. However, it is difficult to obtain long optical path length in optical waveguide for gas-cell with CRDS because the intensity of optical pulse

![Fig. 2. Schematic of ring-down pulse in conventional CRDS system. (a) Without gas. (b) With gas.](image)
becomes small due to the loss of optical waveguide. Hence, the ring-down pulse may be disappeared in optical waveguide with CRDS system before the difference of ring-down time is appeared when the loss of gas-cell part has big loss. Thus, difficulty arises for measuring small portion of gas. To realize gas sensing by using optical waveguide with CRDS, compensating the loss of optical waveguide is desired. Thus, we propose amplified CRDS for compensating the loss of optical waveguide. Moreover, health check will be possible by measuring concentration of CH₄.

Presently we plan to realize integrated device with having “high-mesa waveguide” and “coupler”, which corresponds to “gas cavity” and “mirrors” in conventional CRDS gas cavity, as shown in Fig. 3. SOA (semiconductor optical amplifier) is also integrated on the device to compensate the waveguide loss. Possibly, wavelength filter to cut the ASE (amplified spontaneous emission) from SOA is also integrated on same chip. Of course, we need LD (laser diode) light source (possibly tunable laser diode), as well as PD (photo detector) to measure the plural gas of breath. As we have already discussed, gas cavity and mirrors in the conventional CRDS are possible to be replaced to an optical waveguide and coupler. Here, we use a 9:1 coupler consisting the waveguide loop as an example. 90% of light power goes back to the loop and 10% goes to PD through the coupler. This means that the coupler works like mirror in conventional CRDS of which the reflection corresponds to 90%.

Before realizing such that integrated device, we verify the feasibility of this configuration by using the experiment set-up as shown in Fig. 4. EDFA (erbium-doped optical filter amplifier), wavelength filter, and pigtailed silica high-mesa waveguide are set in the optical fiber cycle. EDFA compensates the total insertion loss inside the optical fiber cycle. Wavelength filter cut the ASE from EDFA. Silica high-mesa waveguide (length is 4.5 cm, Γ_out is designed to be 2.4%) is set in a hermetic chamber with CO₂ inside. This experiment system works like compact amplifier CRDS system as shown in Fig. 3.

![Fig. 3. Schematic of compact amplifier CRDS system](image-url)
4 Results and discussion

To check the feasibility of the set-up explained in chap. 3, we use CO₂ instead of CH₄ in this work because CO₂ has the wavelength of absorption peak near one of CH₄ and can be handled relatively safely. We set the amplification of EDFA to 10 dB to compensate the loss of one optical fiber cycle which is approximately 10 dB due to the insertion loss of the pigtailed silica high-mesa waveguide, wavelength filter and coupler. Fig. 5 shows the results of ring-down waveform without EDFA (a) and with EDFA (b). Wavelength of optical pulse is 1572 nm which is the absorption peak of CO₂ and optical pulse width is 40 ns. As shown in Fig. 5(a), ring-down pulse disappears due to the loss of one optical fiber cycle. However, the number of pulse was enhanced to be four times approximately as shown in Fig. 5(b) because EDFA compensates the loss of one optical fiber cycle. The number of ring-down pulse is over 60, which is equivalent to 10 cm of optical path length in the experiment system. This optical path length is able to measure few 10% of CO₂ because gas absorption is 0.02 dB/cm. We increase the gain of EDFA to obtain more ring-down pulse for lower concentration sensing. It is possible to increase the number of ring-down pulse theoretically but it is difficult to get correct ring-down time due to oscillation because AES generated from EDFA causes oscillation. We need to consider the method to increase the number of ring-down pulses without oscillation in future work.

Fig. 6 shows the waveforms we obtained at different concentration of CO₂ (50% ~ 70%). Blue line is 50% of CO₂. Orange line is 60% of CO₂. Gray line is 70% of CO₂. As shown in the figure, light intensity decreases much faster as CO₂ concentration decreases. This result proves clearly that gas absorption happened at silica high-mesa waveguide which is gas-cell part. Ring-down times and calculated concentration of CO₂ are shown in Table I. The Ring-down times were estimated to be 14.28 µs for the case of 50% concentration of CO₂ and 16.46 µs for the case without CO₂, respectively. This Ring-down time includes no gas-cell part such as optical fiber of the fiber cycle. To calculate the correct concentration of gas, we must convert this time into the time passed through only silica high-mesa waveguide for gas-cell. As the result, ring-down time of with CO₂ and without CO₂ converted time are 9.91 ns for the case of 50% concentration of CO₂ and 11.42 ns for the case without CO₂. By using these ring-down times, we estimated that the concentration was to be 51.29% that was similar to 50%. Similarly, we evaluate
others concentration of CO₂. From these results, the estimated CO₂ were again similar to the actual CO₂ concentrations. The accuracies were within 3% for all the cases, and we have successfully confirmed relatively high accuracy in CO₂ gas concentration. For the actual health check system, it needs to detect 10 ppm order of CH₄ at least [3]. However, the result of 50% CO₂ gas sensing corresponds to the capability of 1000 ppm CH₄ gas sensing because cross section of CH₄ is 1.64 × 10⁻²⁰ cm² (λ = 1651 nm) and absorption cross section of CO₂ is 7.7 × 10⁻²³ cm² (λ = 1572 nm). To accomplish this goal, much lower propagation loss of approximately 0.01 dB/cm with higher Γ_out of 10% is desired. We are still on the way of future improvement.

Fig. 6. CO₂ (50–70%) gas sensing

Fig. 5. Ring down waveform of CRDS. (a) Without EDFA, (b) with EDFA.
5 Conclusion

We have proposed the silica high-mesa waveguide with amplified CRDS for breath sensing. Then, we demonstrated gas concentration (50% to 70% of CO₂) measurement by using silica high-mesa waveguide for the first time. We could successfully confirm that silica high-mesa waveguide could be used for breath sensing rapidly. Further improvement is desired to realize actual compact breath-sensing system.

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<table>
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<tr>
<th>Concentration of CO₂</th>
<th>τ (Without gas)</th>
<th>τ₇ (With gas)</th>
<th>Estimated concentration</th>
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<td>50%</td>
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