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Application of a super-luminescent diode coupled with an interference filter for mixed gas sensing

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Abstract

Laser-based spectroscopic techniques are very attractive methods for trace gas, environmental and industrial monitoring. By employing narrow spectral-width tunable coherent light sources, a single absorption line of the substance to be analyzed can be probed with a laser, avoiding interference signals from other species. Here we report a difference frequency generation (DFG) source in mid-infrared region using a tunable laser (825 – 870 nm) and a fixed wavelength diode laser (1060 nm) in combination with a PPLN having single period. Application of the approach for carbon dioxide (CO_2) monitoring in air was studied. Many gas sensing applications do not require extreme sensitivities up to ppb order and in this case, super-luminescent diodes (SLD) are also of great interest. They are commonly produced in the spectral range of optical fibers telecommunications (1.3 – 1.7 μm), where overtone vibration bands of many gas molecules of interest. As a typical example, measurements of ammonia (NH_3) trace gas were studied for combustion measurement applications. A novel approach of using fiber-coupled SLD along with an interference filter can also be considered for such application to avoid influence of water vapor (H_2O). The interest of this system consists in the possibility to detect several species with a single instrument.

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1. Introduction

There are various applications, such as industrial process monitoring, environmental trace-gas sensing, spectroscopy, where widely tunable coherent light sources operating in the 2-5-μm mid-infrared (mid-IR) region are necessary. Although laser diodes are available, they require low-temperature operation and tuning range is also narrow and discontinuous. On the other hand, difference frequency generation (DFG) based on near-infrared, tunable laser sources can also be considered. Compared with tunable optical parametric oscillators (OPO), the DFG technique has no oscillation threshold and therefore can operate in continuous-wave with low operating power, narrow-band laser diodes or diode pumped solid-state lasers, such as Cr^{3+}:$LiSrAlF_6$ laser.

Recently there is growing interest in quasi-phase-matched (QPM) periodically poled lithium niobate (PPLN) devices for mid-infrared DFG. In the case of quasi-phase-matched DFG process, the first order QPM condition is given by $k_1 - k_2 - k_3 = 2\pi / \Lambda$ [1]. Where k_1, k_2, and k_3 are the wave vectors at the three interacting wavelengths and

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Λ is the PPLN period. This approach offers the advantages of a large effective nonlinear coefficient, noncritical phase matching with zero walk-off, and allows phase-matching with various visible and near-infrared lasers by engineering the PPLN period. A problem with the QPM device is that the tunable range is limited by the period of the crystal domain. On the other hand, PPLN has a wide phase-matching bandwidth around the input wavelength region of 780-900 nm. Therefore using near-IR lasers, such as either a Ti^3+:sapphire laser or a Cr^{3+}:$LiSrAlF_6$ laser (700-900 nm) in combination with a diode laser and a PPLN with a single-period, a wide tuning range should be possible [2]. Here we report a mid-IR DFG source using an external cavity, tunable laser diode (825 – 870 nm), which is termed as a pump laser, and a fixed wavelength diode laser (around 1060 nm), which is termed as a signal laser, in combination with a PPLN having single period. In addition, the feasibility of application of a PPLN-based diode-pumped DFG source to CO_2 gas sensing in air is investigated.

Many gas sensing applications do not require extreme sensitivities up to ppm order and in this case, super-luminescent diodes (SLD) are also of great interest. These semiconductor devices are very compact and reliable light source operating at room temperature and provide a long lifetime. They are commonly produced in the spectral range of optical fibers telecommunications (1.3 – 1.7 μm), where overtone vibration bands of many molecules of interest occur (CO_2, H_2O, NH_3, CH_4…). As a typical example, measurements of H_2O, NH_3 and other trace gas are necessary in combustion applications and for NO_x emission reduction. A novel approach of using fiber-coupled SLD along with an interference filter can also be considered for such application [3]. The wavelength tuning by the interference filter will provide selective gas sensing by absorption spectroscopy technique. The interest of this system consists in the possibility to detect several species with a single instrument. In this research work, an application of the SLD coupled with a solid interference filter is proposed and investigated for NH_3 for combustion studies.

2. Widely tunable difference frequency generation for gas sensing

2.1 Experiment setup

Figure 1 shows the experimental setup. A tunable laser-diode (825 – 870 nm) was combined with a fixed wavelength laser-diode (1060.6 nm) using a dichroic beam splitter. The tunable laser diode with a central wavelength around 850 nm consisted of an external cavity in a Littrow configuration. Coarse tuning from 825 to 870 nm was obtained by rotating the grating and mode-hop free fine tuning range of 16 GHz was possible by a piezo-electric actuator. The fixed wavelength laser was a distributed Bragg reflector type laser diode. Both lasers were

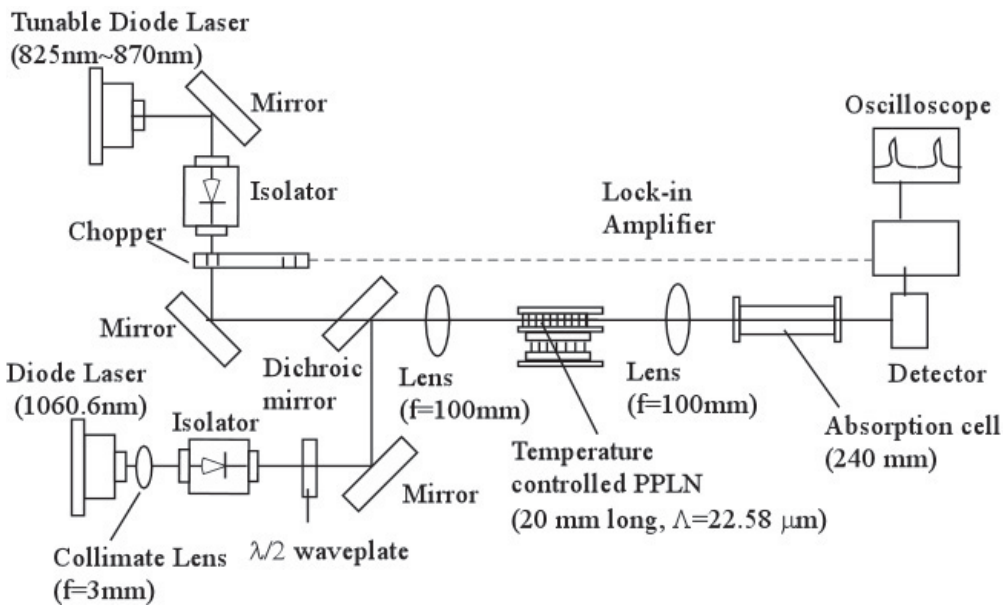

Fig. 1: Experimental setup for a tunable DFG output and gas sensing.

operating with a single-mode output. The laser beams were focused by an achromatic lens (focal length= 100 mm)

at the center of a 0.5 mm thick, 20 mm long, bulk periodically poled $LiNbO_3$ (PPLN) with a domain grating period of Λ= 22.58 μm. The generated mid-IR beam was then focused onto a liquid nitrogen cooled InSb detector (P5172-

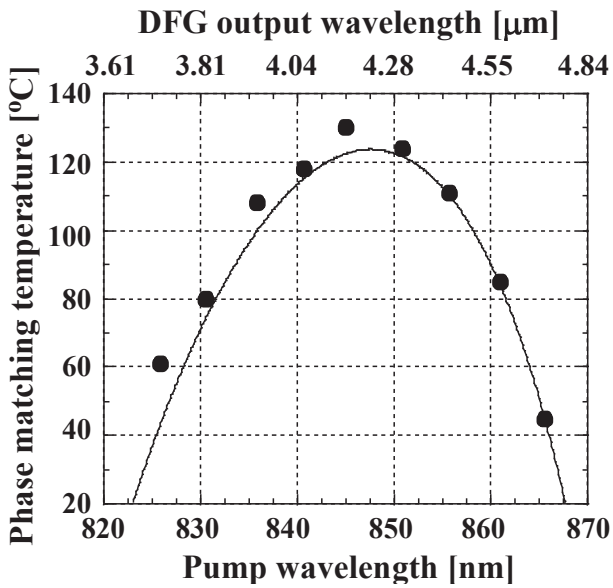

Fig. 2: Tunable DFG output at different pump wavelengths and corresponding phase matching temperature. Signal wavelength: 1060.6 nm and PPLN period Λ=22.58 μm.

100, Hamamatsu Photonics). The measurements were performed with a trigger signal from an in-line chopper rotating at 800 Hz and a lock-in-amplifier combined with an oscilloscope.

In the case of a QPM device, the tunable range is limited by the period of the crystal domain. Nevertheless, PPLN has a wide phase-matching bandwidth around the input wavelength region of 780-900 nm. Due to a broad acceptance bandwidth for phase matching, wide range tuning is expected by altering the PPLN crystal temperature with single grating period, which was not possible with other pump wavelength ranges. Therefore using near-IR lasers, such as either a Ti^3+: sapphire laser or a Cr^{3+}:$LiSrAlF_6$ laser (700-900 nm) in combination with a diode laser and a PPLN, a wide tuning range can be covered [4]. The idler wave generated due to the DFG process was transmitted through a gas sample and absorption studies were conducted by measuring the intensity using the InSb detector. As a sample target gas, CO_2 gas was used and absorption spectroscopy measurements were performed corresponding to the ν_3-band ranging from 2380 – 2275 cm^{-1} (4.20 – 4.40 μm) of the CO_2 absorption spectrum.

Subsequently, for tunable DFG output, the pump laser was tuned and simultaneously the PPLN temperature was altered for a phase-matched condition. Figure 2 shows the tuning characteristics of the DFG output corresponding to the phase-matching temperature. Solid circles show the experimental results and the solid line shows the theoretical calculation based on the QPM condition and the Sellmeier equation [5]. As shown in Fig. 5, a wide tuning range between 3.7 and 4.8 μm was obtained by tuning the pump laser between 825 and 868 nm and altering the PPLN temperature between 20 and 130 °C with single period of Λ=22.58 μm.

The tunable output from DFG process was transmitted through a CO_2 gas sample. Absorption spectroscopy measurements were performed corresponding to the ν_3-band ranging from 2380 – 2275 cm^{-1} (4.20 – 4.40 μm) of the CO_2 absorption spectrum. First, a very weak CO_2 transition near 4.37 μm was considered where minimum absorption was expected based on the HITRAN database analysis [6]. The pump wavelength of 853.3 nm was selected using a wavemeter (Burleigh, model:4500-0) to match the idler wavelength with the 4.4 μm (2389.5 cm^{-1}) having almost 100 times weaker absorption strength (absorption cross-section value of 1.17×10^{-19} cm^2) than that of 4.2 μm (2362.78 cm^{-1}). The pump laser was fine-tuned with a mode-hop free tuning range of about 16 GHz to achieve a tunable DFG output. No distinct absorption peaks due were observed when the idler wave was transmitted through the ambient air. However, when the idler wave was transmitted through a 240 mm long cell filled with a

CO_2 gas pressure of about 0.02 MPa (150 Torr), a distinct absorption peak was observed with the fine-tuning of the pump laser, confirming the absorption due to the ν_3-band of CO_2.

Next, atmospheric CO_2 absorption at a stronger transition at the transition wavelength of 4.2 μm (2366.6 cm^{-1}) was studied by tuning the pump laser to 847.9 nm. The ambient air sample-length was 150 mm. The portion of the absorption spectrum of CO_2 in the ambient air centered at the transition line of 2366.6 cm^{-1} over a range of 0.25 cm^{-1} was shown in Fig. 4. Solid line shows the computed absorption spectrum from the HITRAN molecular spectroscopy database. The spectrum was computed by assuming a Voigt lineshape function at an atmospheric pressure and CO_2 concentration of 360 ppm (parts per million). In the computation, the spectral width of the laser was assumed to be 0.03 cm^{-1} (300 MHz). The dotted line with circles shows experimental measurements by tuning the DFG output. As shown in Fig. 9, the experimentally measured absorption spectrum was slightly broader; nevertheless the absorption measurements were in agreement with the computed absorption-lineshape. In addition, the broad continuous tuning

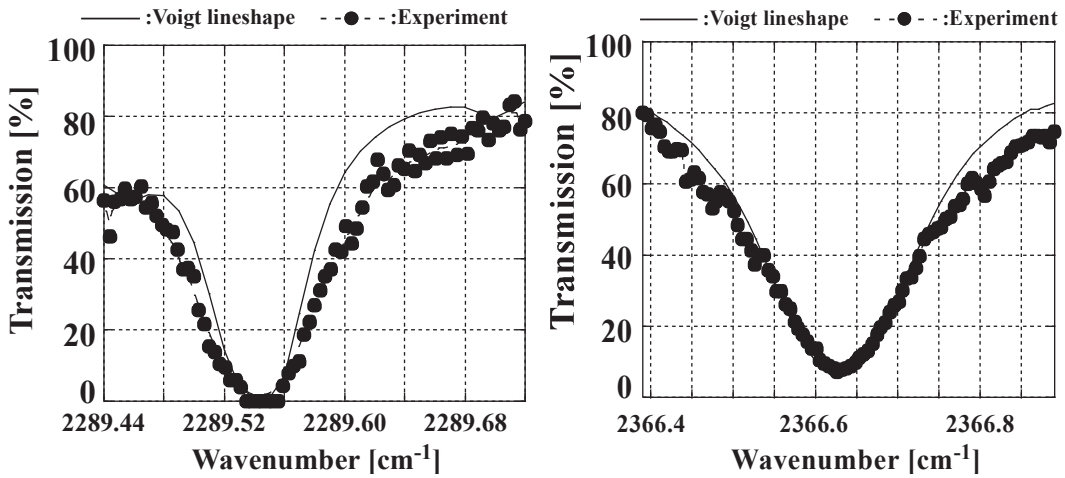

Fig. 3: CO_2 absorption spectra measured with DFG source. (a) 0.02 MPa; (b) CO_2 in ambient air.

range matches with strong fundamental vibration bands of several trace air contaminants, such as carbon monoxide (CO), nitrous oxide (N_2O), formaldehyde (H_2CO), and methane (CH_4).

3. Super-luminescent diode based gas sensing

3.1 SLD based NH_3 sensing

Owing to the great requisite of NH_3 detection in various fields and its rich spectrum in the near-IR region 1500 nm (part of the $\upsilon_1+\upsilon_3$ and $2\upsilon_3$ combination and overtone bands) was selected for analysis which further being the standard for optical communication grade. In the present study, SLD with emission wavelength centered at 1530 nm (EXALOS AG, EXS1510-2111) was used for NH_3 gas measurements. Figures 4(a) and (b) show the proposed schematic and experimental setup for NH_3 concentration measurements using the absorption spectroscopy technique. The collimated output from the single mode fiber-coupled SLD output was passed through a multi-pass cell filled with the test gas, and the beam undergoes multiple number of reflections from the gold-plated surface thereby increasing the effective pass length to subject for trace amount of concentrations. Initially the cell was maintained in vacuum condition whose output serves as the reference. The transmission and broadening effects were studied by varying the concentrations of the test gas maintained in the cell. The absorbed output from the multi-pass cell was analyzed with the help of an optical spectrum analyzer (Yokogawa, AQ6370B).

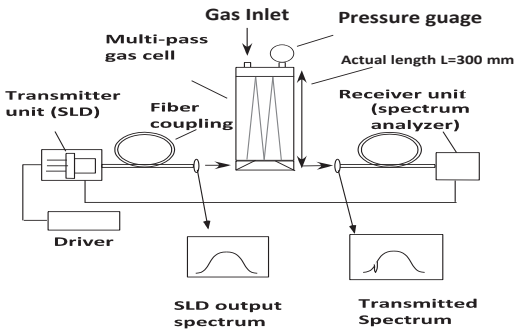

Fig. 4(a): Schematic diagram of NH_3 based SLD sensor system

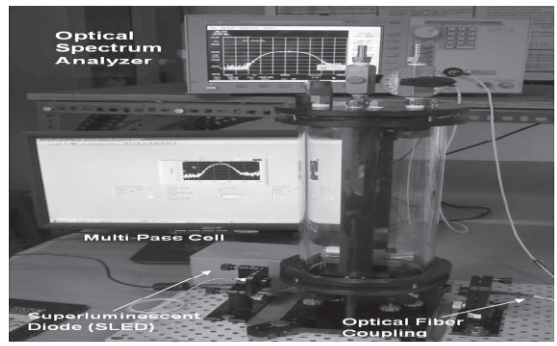

Fig. 4(b): Experimental setup of NH_3 based SLD sensor system

3.2 Transmission spectra

Transmission characteristics were studied for various concentrations of NH_3 in terms of partial pressures of about 15 kPa, 20 kPa, and 30 kPa. Figures 5(a), (b), and (c) show transmission spectra in vacuum condition (black line-reference) and with increased NH_3 concentrations (red line). The measurements were taken at 0.04 cm^{-1} resolution, having an absorption path of 2000 mm. All spectra were recorded at room temperature. It can be seen that with increase in NH_3 concentrations, the transmission intensity reduced indicating that the absorption strength of lines increased with increase in pressure and the absorption lines goes to saturation at high pressures which was evident at 1545 nm region.

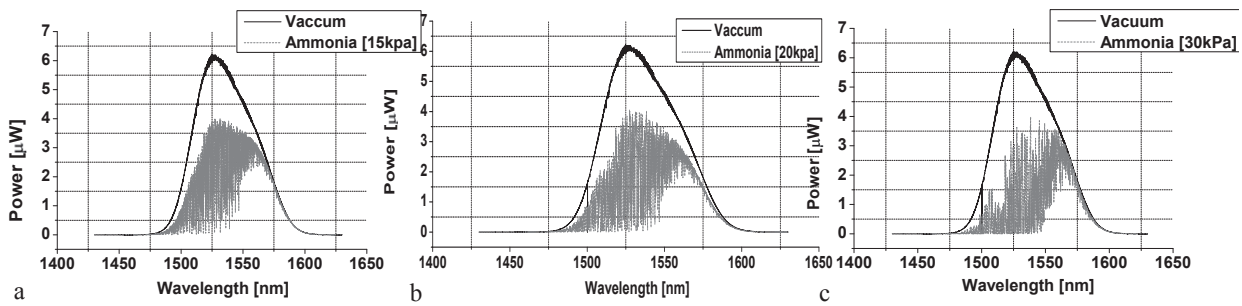

Fig. 5**:** Experimental transmission spectra of 100% NH_3 obtained in vacuum and at 15 kPa, 20 kPa and 30 kPa with a resolution of 0.04 cm^{-1} in an absorption path of 2000mm.

3.3 Estimation of transmittivity

An exemplary has been developed for the transmission of light and can be related to molecular species absorption by the Beer-Lambert law, given by

$$I_t(\lambda) = I_0(\lambda)\exp[-\alpha(\lambda, P, T)L] \quad (1)$$

Where $I_t(\lambda)$ is the transmitted intensity, is $I_o(\lambda)$ the incident intensity, is the $\alpha(\lambda, P, T)$ absorption coefficient at the working wavelength (in cm^{-1}) and is the L absorption path length (in cm). Considering only one absorbing species, the absorption coefficient is given by

$$\alpha(\lambda) = \sigma(\lambda)N \quad (2)$$

Where σ(ν) represents the effective absorption cross-section (in cm^2/molecule) and N the molecular density (in molecule cm^{-3}). The absorption cross-section was computed by considering the absorption line shape represented by

a Voigt profile. In the case of NH_3, the line strength values for the 1500 nm band were estimated from Lundsberg-Nielsen *et al*. [7]. As the SLD has a broadband output spectrum, the effective cross section values for NH_3 was estimated by considering the resolution values of the spectrum analyzer employed during experiments.

Figure.6 displays the experimentally measured (solid-line) and computationally estimated (dotted-line) transmission spectra from 1540 to 1547 nm wavelength region, when the absorption cell was filled with NH_3 at a pressure of 20 kPa. The transmission spectra was normalized in both the cases and plotted without introducing any offset. A good qualitative agreement was observed for line positions in the entire range with those listed in the Lundsberg-Neilson for NH_3. It was evident from these results that the SLD based measurement technique was reliable for the measurement of the concentration of gas species of the order of hundreds of ppm.

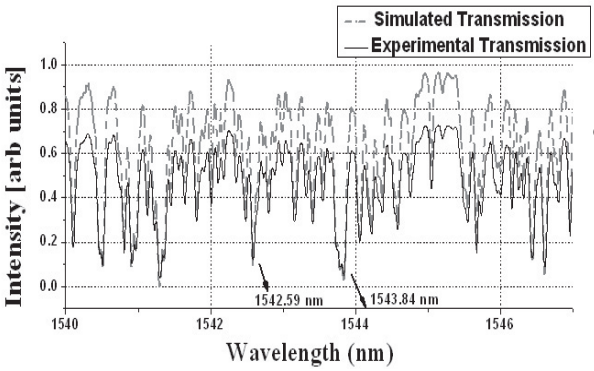

Fig. 6: Displays the NH_3 spectra and compares them with the simulated reference spectra obtained from Lundsberg-Neilson database [7] with the experimental output for NH_3 at a pressure of 20 kPa both at 0.04 cm^{-1}.

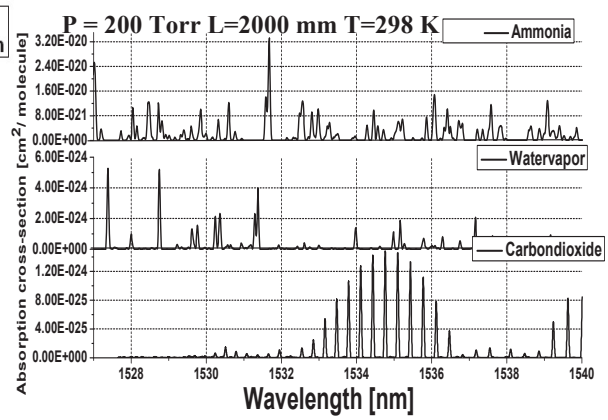

Fig 7: Computed spectra at 20 kPa NH_3, 13% of H_2O, and CO_2 molecules

The broadband nature of the light source enables for the simultaneous detection of multiple gas species. Investigations of SLD based NH_3 gas molecule in 1500 nm region has shown that there is a significant interference of H_2O, CO_2 molecule while detecting NH_3 gas. Figure 7 shows the plot of absorption lines for NH_3, H_2O, and CO_2 molecules in 1500 nm wavelength range based on HITAN database. The line strength values reported in the figure corresponds to a temperature of 298 K. At higher temperature regions such as in combustion analysis CO_2 and H_2O concentrations can be as high as 50% or more where the NH_3 line may get buried. Determining the spectroscopic parameters of each individual gas molecule within these overlapping features thus requires high-resolution spectroscopy and longer effective path lengths which is undesirable. An application of a solid etalon coupled with the SLD was proposed for sensitive and selective (interference free) detection. A solid etalon can be placed along the path of the light transmission and can be tuned to appropriate absorption lines. Figure 8 shows vibrational

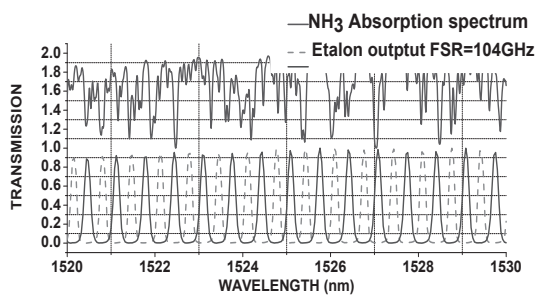

Fig.8: Ro-vibrational absorption lines of NH_3 and multiple longitudinal modes of etalon showing different instantaneous cavity length scan.

absorption lines of NH_3 and longitudinal modes of the etalon based on its free-spectral range for different instantaneous scan lengths. The solid etalon can be fine-tuned to one of the selected absorption line by tilting the etalon and changing the optical path length. This detection technique could be a practical alternative to DFB diode lasers for in situ detection of a relatively high level of H_2O, NH_3 concentrations, over 1 wt% such as in the combustion processes. The broadband nature of the light source of SLD coupled with the solid etalon should also enable the simultaneous detection of multiple gas species with selectivity.

4. Conclusions

A compact, widely tunable coherent optical source based on a difference frequency generation technique in a quasi-phase-matched PPLN crystal pumped by two single-frequency laser diodes (825-870 nm and 1060 nm) for the detection of various gases was developed. The difference-frequency output was tunable from 3.7 to 4.8 μm with a single PPLN period Λ= 22.58 μm. The tuneable DFG source was used for absorption measurement of CO_2 in open air and it was possible to use the source for the detection of ambient CO_2 based on the absorption spectroscopy. The use of fiber-coupled pump and signal lasers should allow a compact and rugged configuration.

In addition, super-luminescent diode (SLD) based gas sensing technique was developed and demonstrated for NH_3 gas. Mixed gas sensing based on a broad-band SLD (1535 nm) coupled with an external etalon was proposed. The wavelength tuning by tilting the etalon will provide selective gas sensing by absorption spectroscopy technique.

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