All-telecom diode laser based mid-IR source for spectroscopic detection of HF, H₂O and HDO

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Abstract: A novel difference-frequency mixing architecture for coherent generation of tunable mid-infrared light is reported. Two CW single-frequency diode laser pump sources operating at 1.56 and 0.98 μm were mixed in a periodically poled LiNbO₃ crystal and generated 0.25 mW of tunable mid-infrared light at 2.64 μm. The performance of this new source was demonstrated by the spectroscopic detection of HF and water isotopes H₂¹⁶O, H₂¹⁷O, and HD¹⁶O at various reduced pressures. Using direct absorption spectroscopy, a peak-to-peak noise equivalent absorbance of ~1E-4 was observed (0.6 s integration time), corresponding to a HF detection sensitivity of 12 ppb/m at a sampling pressure of 50 Torr.

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1. Introduction

Tunable mid-infrared laser sources are increasingly utilized for rapid precision gas sensing in environmental, industrial, and research applications. For this purpose, advanced compact solid-state laser sources, including quantum-cascade-, lead-salt- and difference-frequency generation (DFG) -lasers have been developed. Primarily, the developments of these sources were aimed at wavelengths covering the 3-5 μm and 6-16 μm region, overlapping with strong fundamental rotational-vibrational bands of many trace gas species [1]. Access to another prime spectroscopic window ranging from 2 – 3 μm has been limited due to the difficulty of producing tunable laser sources in this wavelength region. This spectral region offers access to unique absorption bands of HF (S=1E-18 cm molecule⁻¹), H₂O with respective isotopes, NH₃ (free of any water interference), and CO (2.3 μm)[2,3]. This paper describes the design and evaluation of an all telecom diode laser based mid-IR source at 2.64 μm, which can also be configured to operate in the 2.3 – 2.7 μm wavelength range. This source was then used to measure HF and H₂O in a closed cell as means of demonstrating the narrow laser linewidth, and superior amplitude and frequency stability.

2. Experimental

The use of standard telecom diode lasers as DFG pump sources offers robust fiber optic coupling while providing the best possible pump beam quality (i.e. Gaussian), and a wide wavelength coverage by selecting and multiplexing off-the-shelf near-IR diode laser channels. In addition, telecom diode lasers based on InGaAsP/InGaAs/GaAs typically possess similar temperature tuning coefficients as one another (~0.4 cm⁻¹K⁻¹). This offers predictable spectral performance and leads to a self-compensation of wavelength drifts in a DFG-based mid-IR architecture and minimizes the effect of electronic driver temperature instabilities induced by environmental changes. This property can significantly increase the spectroscopic stability and hence the accuracy of gas concentration measurements. In particular, if such a mid-IR source is applied to operate in industrial applications where considerable changes of temperature, pressure and vibrations are encountered.
Fig. 1 shows the optical architecture used in this experiment. A 1562 nm single-mode fiber pigtailed DFB diode laser serves as the DFG pump source and provides an optical fiber output power of 15 mW, with <2 MHz linewidth and a 46 dB sidemode suppression ratio. The output fiber is fusion spliced to an Er/Yb optical fiber amplifier and produces a maximum output power of 575 mW. The long-term power stability of the seeded amplifier was measured to be less than 0.2 % h⁻¹.

![Diagram of optical architecture](image)

The DFG signal source is an optically pumped semiconductor laser (OPSL™) and has recently become commercially available (Coherent OPSL™ 980-500) [4,5]. The OPSL™ was designed for use as a telecom pump source in Er³⁺ doped fiber amplifiers. Unlike other high-power 980 nm telecom pump diode laser sources, this laser provides excellent spectroscopic characteristics of >500mW single-frequency laser radiation from a single mode fiber (Corning 1060 PureMode™, Mode field diameter = 5.9 μm @ 980 nm). Both fibered laser sources were fusion spliced to a WDM. The output fiber of the WDM was terminated by a Flexcore 1060 single-mode FC-APC fiber (8° fiber polished end). Respective insertion losses of 11% (OPSL) and 5% (Er/Yb fiber amplifier) were introduced by fiber fusion splices to the WDM, and inherent WDM coupling losses. The two DFG pump beams were imaged (M~12) into a 19 mm long / 0.5 mm thick PPLN crystal with a 28.7 μm quasi-phasematching period. The PPLN crystal was AR coated with a single SiO₂ layer and optical transmissions at 982 nm and 1562 nm were measured to be 0.84 and 0.98, respectively. A plano-convex CaF₂ lens (f=50mm) was used to collect and collimate the DFG radiation. Residual pump beams were blocked by a Ge-filter.

For spectroscopic measurements, the DFG beam was directed through a 10 cm long gas-probing cell and the absorption signals detected with an InSb detector. For safety reasons associated with handling HF gas-mixtures, the cell was made from Teflon material (O.D.=1 in. I.D.=0.5 in.), and end-fitted with sapphire windows sealed with Viton o-rings. We employed the filling line depicted in Fig.1b for filling the gas cell with HF dry N₂ mixtures at various pressures (1 – 300 Torr). All tubing was made of ¼” O.D. stainless steel or glassware. For safety reasons, the gas filling line was operated under a venting hood and the exhaust gas pumped through a liquid-nitrogen trap to avoid HF contamination of the vacuum pump.

3. Optical characterization and spectroscopic measurements

Before splicing the OPSL™ device to the WDM, the output power as a function of pump current was measured. At a device temperature of 25 °C, a maximum optical fiber output power of 611 mW was observed with a pump current of 2.2 A. With various fiber and achromatic lens insertion losses a pump power of 492 mW (20% total insertion power loss) was available at the input facet of the PPLN crystal. Likewise, insertions losses in the 1562 nm channel resulted in a maximum effective pump power of 440mW (15% total insertion power loss) at the PPLN input facet. A maximum DFG power of 250 μW was measured, which corresponds to a conversion efficiency of 650 ± 25 μW/W² cm⁻¹. The conversion efficiency was determined by measuring the slope of DFG power (past the Ge-filter) at different input
power levels. Both pump and DFG power levels were measured with a NIST traceable calibrated thermopile detector.

The mid-infrared radiation could be varied over 7.59 cm\(^{-1}\) (6404.98-6397.39 cm\(^{-1}\)) by means of temperature tuning the 1562 nm DFB DL from 19.31 °C- 38.65 °C. Within this tuning range the Hitran database [1] lists molecular absorptions of \(\text{H}_2^{16}\text{O}, \text{H}_2^{17}\text{O}, \text{H}_2^{18}\text{O}\) and \(\text{HD}^{16}\text{O}, \text{HF}\) and \(\text{NO}\). For the detection of \(\text{HF}\) and \(\text{HDO}\) at 2.64 \(\mu\)m, the DFB-DL was temperature tuned to 6398.599 cm\(^{-1}\) (T\(_{\text{DFB-DL}}\)=35.56 °C) and current modulated by applying a 160 Hz triangular waveform. Fig.2a shows the respective direct absorption spectra averaged over 313 s. The mid-IR scan was frequency calibrated using a Ge-etalon (FSR=0.01601cm\(^{-1}\)). An analysis of the Ge-etalon fringe spacing showed a continuous frequency tuning (\(-1E-3\text{ cm}^{-1}\text{ pt}^{-1}\)), superimposed by a small 1st order nonlinearity of 8E-6 cm\(^{-1}\text{ pt}^{-1}\) (500 pt/scan). This nonlinearity was found to be constant over the DFB DL temperature tuning range, whereas the tuning rate showed a temperature dependence of 8.57E-6 cm\(^{-1}\text{ pt}^{-1}\text{ °C}^{-1}\).

![Absorbance Graph](#)

*a)*

![Frequency Graph](#)

*b)*

Fig.2: a) Direct absorption spectra of \(\text{HF}\) and \(\text{HDO}\) at 2.64 \(\mu\)m (p<10 Torr). The residual peak-to-peak baseline noise is <0.01%. b) Spectroscopic stability in units of cm\(^{-1}\) and MHz over a 200-minute time period. The peak fit precisions were better than ±4 MHz and are represented as solid squares in the upper graph 2b).

To evaluate the long-term spectroscopic stability, HF absorption spectra at 2.64 \(\mu\)m were periodically measured over a 200-minute period. Fig. 2b depicts the frequency drift shown both as relative frequency within the scanning width and offset frequency. Upon enabling laser operation, a maximum DFG-frequency drift of ±60 MHz was initially observed and this gradually decreased to ±20 MHz over a 2 hour period. At a later time, the drift was less than a 5 MHz over 10 minute periods. Occasional mode-hopping of the OPLSTM was observed when the laser was operated from a cold start. No mode-hopping was observed after the OPLSTM was continuously operated for 2-3 hours and fully temperature stabilized. In summary, a novel tunable DFG based mid-infrared spectroscopic source at 2.64 \(\mu\)m and its application for the detection of \(\text{HF}\) and \(\text{H}_2\text{O}\) was demonstrated. Replacing the Er/Yb fiber amplifier with an Er\(^{3+}\) doped fiber, inline with the OPLSTM and DFB-DL at 1.5 \(\mu\)m, will result in an ultra compact low cost design [6].

References:
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