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Photoacoustic gas sensor based on an array of quantum cascade lasers and a quartz tuning fork: towards multi-gas sensing

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Abstract: We present a photoacoustic gas sensor employing QCLs and a versatile detector based on a quartz tuning fork and an acoustic radial resonator. We present first measurements and perspectives for multi-gas sensing. © 2023 The Author(s)

1. Motivation

Multi-gas detection within a single instrument is a versatile and cost-effective solution. However, detecting molecules presenting a broadband absorption spectrum at trace level while keeping a good selectivity is a challenge for optical methods.

Photoacoustic spectroscopy (PAS) is a laser based detection method that might be able to meet these requirements. It allows to selectively detect molecules at concentrations down to parts per billion (ppb). Moreover, the detection is wavelength independent thus enabling a strong multi-gas capacity [1].

In PAS, the acoustic signal can be detected by quartz tuning forks (QTF) [2]. The first and most common QTFs used in PAS are watch QTFs, due to their availability, small size (4 mm x 2 mm) and extremely low cost (<1 \$). To enhance their sensitivity, acoustic longitudinal resonators are used, generally taking the form of tubes of sub-millimeter diameter and 1 cm length placed around the QTF and in which the laser beam passes [3] (see Fig. 1). This configuration called "on-beam" allows to reach state-of-the-art sensitivity, quantified by a normalised noise equivalent absorption (NNEA) of $\sim 1 10^{-9}$ cm⁻¹.Hz^{-1/2}. However, focusing the laser beam inside those tubes is difficult, strongly degrading the instrument multi-gas capacity, which should be the main advantage of PAS.



Fig. 1. (a) Scheme of a watch QTF in "on-beam" configuration. (b) Scheme of our "in-plane" configuration. (c) Acoustic simulation of the resonator radial mode (d) Sensor prototype including a gas hermetic cell, coupled to a QCL array inside a metallic box, and control electronics.

To circumvent these drawbacks we presented a new configuration based on a custom QTF (cut, etched and electro-plated in our clean room facilities) coupled to an acoustic radial resonator [4]. The careful design of this new detector enabled a compromise between the sensitivity, reaching 3.9 10^{-9} cm⁻¹.Hz^{-1/2}, and an easier laser beam focusing, in a $\sim 2x2$ mm² area, thus relaxing alignment constraints compared to the classic configuration. Here we present an improvement of this configuration (see Fig. 1), and its first coupling to a quantum cascade laser (QCL) for measuring NH₃ with perspectives for multi-gas sensing.

2. Our work

The previous configuration [4] was first optimised through finite element simulations by reducing acoustic losses of the acoustic resonator and minimising the sensitivity by changing the laser beam into "in-plane" configuration

(cf. [2]) compared to the QTF.

We then designed and fabricated this new configuration as shown in Fig. 1. A first characterisation was made using a distributed feedback telecom diode demonstrating a near tenfold sensitivity enhancement to reach 5 10^{-10} cm⁻¹.Hz^{-1/2} compared to the previous configuration. In a second time, we coupled our sensor with a laser array, comprising 9 distributed feedback QCLs emitting 2-3 mW each between 1040 and 1130 cm⁻¹ (see Fig. 2). The lasers beams were collimated by means of flat InP micro-lenses and sent into the gas cell, on top of the gap between the QTF prongs. A double pass was implemented thanks to a miniature gold mirror placed at the back of the cavity. We then characterised the sensor by using only one QCL targeting NH₃. The QCL was operated in quasi-CW for lower consumption and better thermal management: it emitted 10 ns large pulses, at a duty cycle of 9 % and at a 9 MHz frequency. Wavelength modulation (WM) at the resonator frequency (21,424 Hz) was implemented by modulating the lower current with a sine wave whose amplitude was optimised to maximise the PAS signal. A certified mixture of NH₃ diluted at 200 parts per million (ppm) in N₂ was used to flush the cell at a flow rate of 0.1 L/min. The QTF coupled to the radial acoustic resonator was placed inside a stainless steel gas cell with its signal amplification electronics and demonstrated a frequency of 21,424 Hz and a quality factor of 1,600, reduced compared to the bare QTF due to the coupling between resonators. The signal was fed to a commercial lock-in amplifier.



Fig. 2. Left: Mirsense's QCL array and FTIR characterisation. Right: Measured spectrum of NH₃ by varying the QCL temperature. Inset: Overlapping Allan-Werle deviation of the signal.

The spectrum was measured by slowly sweeping the QCL temperature. The absorption line of NH₃ at 1049.5 cm⁻¹ was found at a setpoint temperature of 23 °C (see Fig. 2).

After a careful optimisation of the laser beam position and coupling of the resonators, we obtained a signal of 78 mVrms, while the background was around 3 mVrms. The noise was measured to be 0.24 mVrms by computing the signal Allan-Werle deviation for an integration time of 2 s (see Fig. 2). A signal drift appears for integration times longer than 10 s which is still under investigation (recent measurements demonstrated a turning point around 70 s), as the intrinsic stability of the sensor is much better. From these values one can obtain a sensitivity of 2.2 10^{-9} cm⁻¹.Hz^{-1/2} which is close to the state-of-the-art. To conclude, we developed a new photoacoustic gas sensor providing a good compromise between the sensitivity and the multi-gas detection capability. This sensor was characterised for the first time with a QCL emitting around 9 µm targeting NH₃. A sensitivity close to the state-of-the-art was obtained, paving the way towards multi-gas and cost-effective sensors.

In the future, we will characterise this detector with the full array of QCLs, on several NH_3 lines and on molecules with broader absorption spectra. Integration of several QCL arrays into a single and compact instrument is also under way.

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