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Gas Detection with a Micro FTIR Spectrometer in the MIR Region

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Abstract

We report on the application of a silicon micromachined lamellar grating interferometer in a Fourier Transform Infrared (FTIR) spectrometer for the detection of gases in the mid-infrared (MIR) region. The FTIR spectrometer was equipped with MIR optical fibers for light coupling. Gas measurements in the MIR region were focused on specific gases (CO2, CH4) in order to determine the limit of detection and the selectivity that could be obtained using such a micro-spectrometer. The influence of humidity on the gas response has also been investigated. Using two different spectral regions we were able to detect concentrations of carbon dioxide over a span from 100 ppm to 9000 ppm with a theoretical detection limit evaluated at 10 ppm. The performed gas measurements showed the proof of principle of a miniaturised FTIR gas analyzer based on a micromachined spectrometer.

Keywords: Spectroscopic gas sensing, miniturized FTIR, MEMS, mid infrared, green house gases

1. Introduction

The current FTIR systems are bulky and expensive and therefore their use is often limited to laboratories. The developments of a micro FTIR gas analyser, having a smaller size and at a lower cost would enlarge the fields of application of such systems. Most compact spectrometers use the dispersive effect of a grating. A novel MEMS-based Fourier spectrometer based on a lamellar grating interferometer has been realized on silicon [1]. We have demonstrated before its application for gas detection in the near-infrared region [2]. Here we report on the adaptation and the use of the spectrometric module for gas measurements in the MIR region.

2. Features of the micromachined spectrometer

2.1. Operational principle of lamellar interferometer

A lamellar grating spectrometer has the specificity that it is a common path interferometer and the wave front is split spatially and not in intensity by a beam splitter as in conventional Michelson systems. It usually setup in

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reflection as shown for the far infrared [3, 4, 5] and other millimeter wave regions [6]. This gives the possibility to realize very compact interferometer modules if the driving mechanism can be miniaturized too. One demand is to keep the size of the active area of the lamellar grating as large as possible and realize a small period in order to have large separation angles of the diffracted beams. As a good compromise a period of 100 to 300 micrometer seems convenient for near infrared and mid infrared interferometer systems [1, 7]. Smaller periods below 50 microns might be adapted for particular applications at shorter wavelengths [8, 9].

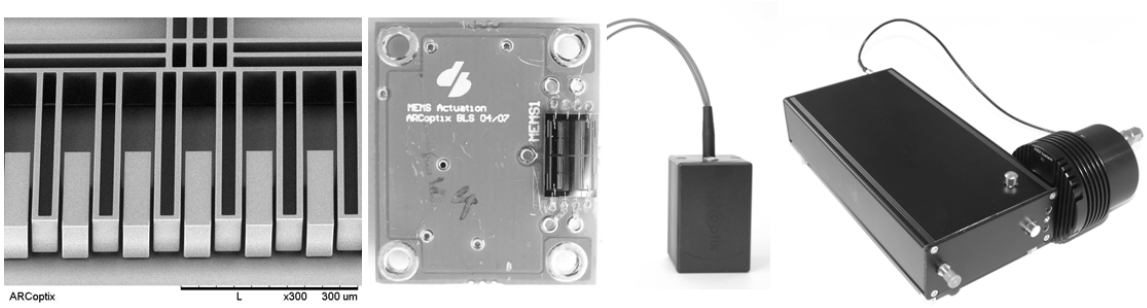

Fig. 1 The building principle of the micro FTIR module. A MEMS chip in a) is used as the core of the interferometer, mounted on a PCB as shown in b) and hermetically packaged in housing comprising optical components for illumination management in c). A detector and electronics are embodied to complete the spectrometer in d).

2.2. Fast scanning MEMS spectrometer characteristics

The heart of the lamellar MEMS based Fourier transform spectrometer FTS used in our experiments is a Si chip of 5 time 3 mm^2 size. It contains a lamellar grating arrangement as seen in the Fig. 1a) with a period of 100 microns and comb drive actuators. This MEMS scanner with an active area of 75 x 3000 μm^2 is bonded on a PCB as seen in Fig 1b). Mirror optics is used to focus the light from the entrance onto the MEMS active area and couple the modulated light back into the output. Light is injected via fluorinated fibers of 100 µm diameter from IRphotonics. MEMS chip and optics are incorporated in a hermetically sealed interferometer module of 55 x 30 x 30mm^3 shown in Fig. 1c).

The common path design allows to use only one optical element for collection and focusing of light. The MEMS scanner operates at 200 Hz and allows up to 300 µm path difference corresponding to approximately 20 cm^{-1} resolution. A detector, the electronics for driving and data acquisition are integrated into a slim aluminum housing as seen in Fig. 1d). The resonance operation is typical for MEMS movement as it assures a reliability of the scanning mechanism that is otherwise only found in laser controlled interferometers. Typical resonance frequencies are above 200 Hz and usually 3000 measurements points are recorded which leads to measurement times of 2 µs per point. A high speed detector has to be used that has reduced noise performance. We used a MCT detector from VIGO with sensitivity between wavelengths of 2 and 5 µm. The detector is thermoelectrically cooled down to -70°C. Due to the short measurement time the signal to noise ratio (SNR) of the system for a single scan is too low for most applications. The conventional method is to change the scan speed which cannot be applied in our case because of resonance driving. The SNR can be improved only by averaging signals. We used averaged signals and as expected the noise level is decreased as the square root of the number of averages. The instrument performance limits are reached at 30000 averages and the SNR does not improve any more. A reasonable performance is obtained if 1000 interferograms are averaged. This takes about 5 s and gives a signal to noise ratio of better than 1000 for signals between 2.0 and 3.5 µm wavelengths.

3. Gas sensing application

3.1. Sensing setup and components

A conventional gas sensing setup was used comprising a multipass White-cell [International Crystal Laboratories], a halogen lamp, optical fibers and some collimation optics with CaF lenses. The 0.5 l White-cell has a maximum path lengths of 6 m. Different light sources were tested. Results presented here are obtained with a stabilized 50 W halogen lamp from Jobin-Horiba Yvon. Controlled gas flow was provided by automated system installed at the Institute of Physical and Theoretical Chemistry, University of Tübingen, Germany. It gave the possibility to control gas flows and humidity over wide ranges of concentrations and make multi-gas experiments.

3.2. Application to green house gases

The spectral sensibility and the signal to noise ratio limits the gases that could be measured. Within the measurement range are the typical green house gases: carbon dioxide CO_2, methane CH_4, nitrogen oxides NO_2 and water vapor. We used the following signatures accessible in our experiment. Carbon dioxide CO_2:2.67 μm (relative strengths 1), 4.25 μm (relative strengths 10), Methane CH_4 3.28 μm, Water vapor H_2O 2.67 μm. Typical transmission spectra for different gases are shown in Fig. 2. One recognizes that the methane spectra contain still water which is due to adsorption at the side wall of the white cell and insufficient purging in successive concentration measurements. The limited resolution of the instrument does not allow identifying the fine structure of the absorption peaks.

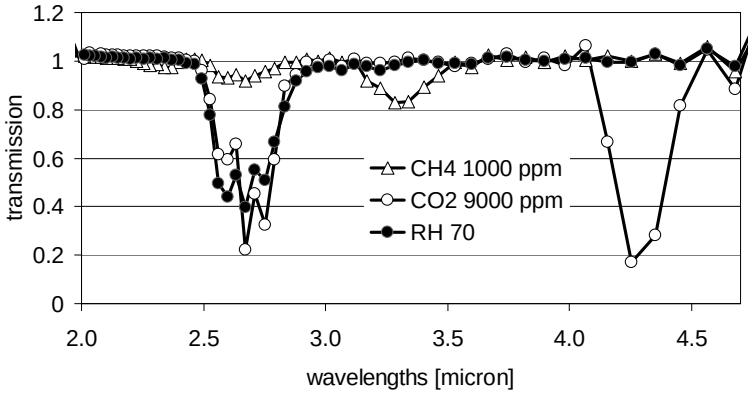

Fig. 2 Transmission spectra for green house gases recorded with the miniaturized FT analyzer setup.

We conducted two types of experiments. We measured single gas concentrations and concentrations in mixtures of several gases. The experimental setup did not allow heating up the white cell to prevent from adsorption. Purging with synthetic air was used to clean the measurement chamber. This procedure is not perfect and leads to measurements errors due to gas capture in water condensation at the inner surfaces of the White cell. The purge time was set to 10 min for the 0.5 l cell volume at a flow rate of 400 sccm (standard cubic centimeter per minute). We measured the absorption of light as a function of the concentration in parts per million (ppm) at a single wavelength of 2.67 μm for CO2. The absorption peak at 2.67 μm is about 10 times less strong than the absorption at 4.25 μm and therefore suited for measurement of high gas concentrations. Absorption values higher than 1 are not anymore accessible with the current system. At 4.25 μm the dynamical range is reduced to the detector performance and the light transmission of the fibers. The maximum absorption value measurable with the system is reduced and only very low concentrations below 50 ppm are accessible. To show the performance of the system, we plot in Fig. 3 transmission and absorption measurements when the concentration of CO_2 is changed between 100 and 200 ppm. It

is clearly proofed that variations larger than 10 ppm can be detected. Considering the possibility of measuring at several wavelengths and using chemometrical analysis the high potential of this technology becomes visible.

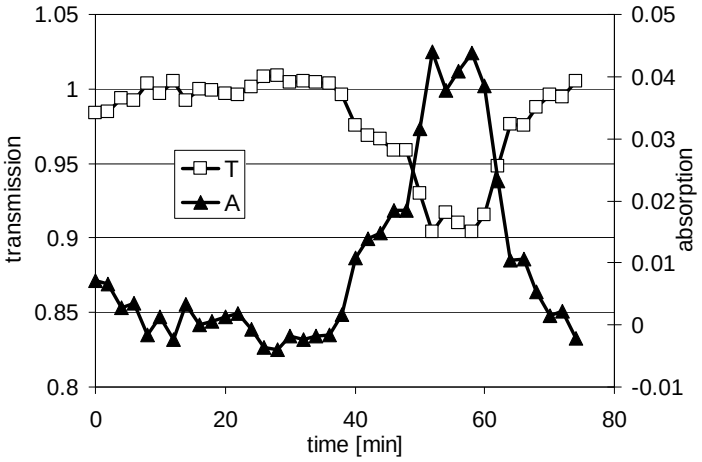

Fig. 3: Transmission (T) and absorption (A) at 2.67 µm for time varying concentrations of CO2. Concentration was changed between 100 and 200 ppm.

4. Conclusion

A multi-wavelengths gas analyzer was set up using FTIR spectrometer with a micromachined lamellar grating. The performance of the system with a long path cell of 6 m approaches today standards. With a larger spectral range and multi-wavelengths detection new possibilities for miniaturized multi-gas sensing will be opened.

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