Plasmonic enhanced CMOS non-dispersive infrared gas sensor for acetone and ammonia detection

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***Abstract*—This work presents novel silicon-based plasmonic infrared emitters that can be used in non-dispersive infrared (NDIR) gas sensors for acetone and ammonia detection. Acetone is of interest for its increasing importance in air quality monitoring and diagnosing diabetes through breath analysis. Detection of ammonia is important due to its broad and diverse range of roles in various environmental processes and industrial technologies. The devices are designed to emit narrowband infrared radiation at the desired wavelengths of 8.26 µm and 10.6 µm for acetone and ammonia detection, respectively, and are relatively inactive at others wavelengths. They have the advantages of high selectivity, low power, low cost and are CMOS compatible. The silicon emitters were simulated using COMSOL, fabricated in a commercial foundry, and then tested on a gas bench rig in both dry and humid conditions. Our simulated and experimental results correspond well with each other and both sensors can detect acetone and ammonia in the concentration range from 50 ppm to 200 ppm and from 10 ppm to 20 ppm, respectively. The designed plasmonic NDIR is also integrated into a portable gas sensing unit.**

Keywords—acetone, ammonia, infra-red, CMOS, NDIR, plasmonics, portable gas sensor unit

I. INTRODUCTION

Acetone is a Volatile Organic Compound (VOC) that can be found naturally in the environment, human body and can also be produced in industry [1]. Inhalation and exposure to acetone could cause acute irritation and minor neurological disturbances [2]; therefore, it is an important substance for air quality monitoring. Elevated levels of acetone can also be found in human breath, which could be a warning sign for diabetes [3]. Ammonia is another natural gas that can be emitted by human activity e.g. in chemical and automotive sectors. Exposure to ammonia gas can cause serious health problems for humans and ecosystems, even at low concentrations in range of tens of ppm [4]. In medical testing, significantly higher breath ammonia levels are associated with chronic hepatitis and liver failure [5]. The recommended exposure limit for acetone is 200 ppm [1] averaged over a 10-hour work shift and 50 ppm for ammonia [6] averaged over an 8-hour work shift, and these are the maximum testing concentration levels of both gases presented in this work.

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There are various approaches for both acetone and ammonia detection, including sensors based on metal oxide technology [7, 8] and acoustic waves. Non-dispersive infrared (NDIR) sensing is chosen for this application because it is a physical technique and has been successfully used for the detection of CO_2 in breath and indoor air [9, 10]. To date few researchers have dedicated their attention to the detection of acetone and ammonia [11] using this technology. Compared to metal oxide gas sensors, NDIR sensors are more stable and suffer less from baseline drift/ageing, and hence have superior repeatability over the longer term. They also have the advantage of higher selectivity and less cross-sensitivity to humidity [12]. The IR sensor is based upon the simple Beer-Lambert absorption law [13]:

$$I = I_o e^{(-\alpha l)} \quad (1)$$

where I is the light transmitted through the sensor cell, I_o is the light incident on the sensor cell, l is the cell length (optical path length), and α is the absorption coefficient of the target gas.

A typical sensor, as presented in Fig .1, comprises a light emitting source, an optical filter and a detector, all housed inside a chamber unit. Commercial NDIR gas sensors are generally expensive (> $100), bulky and can only detect concentrations at percentage level. In order to improve the sensor to detect low ppm levels with a faster response at low cost, a new approach needs to be explored.

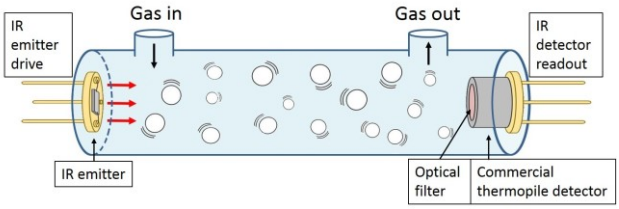

Figure 1: Basic structure of NDIR sensor

As different gas molecules absorb infrared at different wavelength, the IR emitter can be tuned at specific wavelength. Therefore, we investigated a next generation NDIR gas sensor with plasmonic based IR emitters, which can emit a narrow band infrared radiation at low power, to improve both sensor sensitivity and selectivity. Due to the presence of the plasmonic structure, the device has the potential to discriminate different gases at specific wavelengths and this could enable removing

the relatively large optical filter from the sensor chamber [14]. Tungsten is used as a standard CMOS fabrication material which will significantly reduce the cost of the devices. Our work on plasmonic structures for enhanced IR gas sensors has resulted in

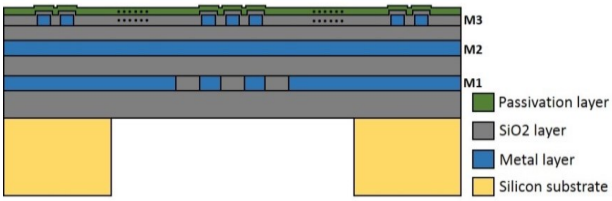

Figure 2: CMOS MEMS emitter cross section with micro-structured plasmonic metal 3 layer M3 and embedded heater M1 (not to scale).

a patent application [15].

II. METHOD AND SIMULATION

Previously, a CMOS based emitter design without a plasmonic layer has been investigated [16]. It consisted of a micro-hotplate device with CMOS metal layer embedded in a dielectric membrane [17]. Based on that, a patterned plasmonic layer has been added [18] to increase the radiation intensity almost 4-fold compare to the non-plasmonic design at a desired wavelength [19]. It is a three metal layer structure as shown in Fig. 2. Metal one (M1) layer is a resistive microheater, M2 is a heat sink for thermal uniformity and M3 (top layer) is a periodically patterned extruded cylindrical islands design for plasmon resonance. The design was fabricated in a commercial foundry using bulk technology. The membrane structure, where a resistive microheater is embedded, is fabricated via a post CMOS deep reactive-ion etching (DRIE) process, which both mechanically supports and thermally isolates the heater from the sidewalls. The top passivation layer is made of silicon nitride.

The periodic pattern in the plasmonic layer composed of arrays of round extruded islands where their parameters that is diameter and spacing control the resonance frequency, which

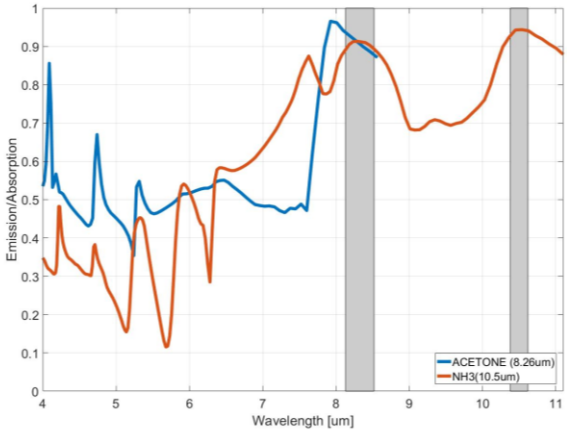

Figure 3: COMSOL simulation results for acetone (max peak at 8.26 µm) and ammonia (max peak at 10.5 µm) target gases, shaded areas represent IR filter bandwidths (0.26 µm and 0.18 µm, respectively).

defines the infrared emission peak. In our designs the period and diameter were tuned to provide enhanced infrared emissivity at specific wavelength for the target acetone and ammonia gases which uniquely absorb IR radiation at 8.26 µm and 10.6 µm, respectively. In order to find the optimum design of plasmonic structures for the target gas, the model was initially built in COMSOL v5.1 Multiphysics software for simulation. Different ratios between period and diameter were trialled with 5% manufacture variance using a finite element method to generate the desired frequency spectra. The best performance for acetone detection was obtained with a surface structure of diameter and period at 3.1 µm and 7.6 µm, respectively. Fig.3 shows the COMSOL simulation results with the shaded area indicating the IR filter with 0.26 µm bandwidth. The simulation results show a close to black body radiation at the desired wavelength centred at 8.26 µm and a relatively low emission at other wavelengths. Similarly, the best performance for ammonia detection was obtained with a surface structure of diameter and period at 3.6 µm and 10.8 µm, respectively, which was also confirmed by the simulation results presented in Fig. 3.

Scanning electron microscope (SEM) images of the fabricated emitter chip for acetone are shown in Fig. 4 and Fig. 5. Our plasmonic structure designs have a hexagonal lattice arrangement as shown in Fig. 4a and the height of the extruded cylindrical structure is fixed at 0.5 ± 0.1 µm as presented in Fig. 4b. As shown in Fig. 5, the centre circle is the microheater with the plasmonic structure layer, both embedded in the membrane (outer circle).

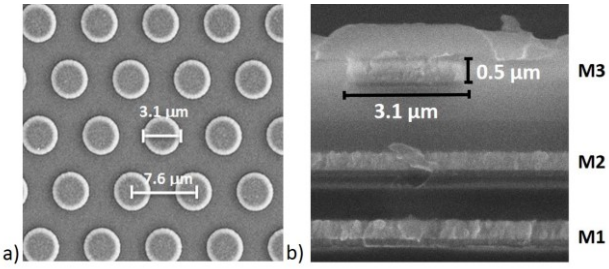

Figure 4: a) Magnified SEM image of plasmonic structures metal 3 layer (top view), and b) magnified SEM image of the cross section of the emitter chip tuned for acetone detection.

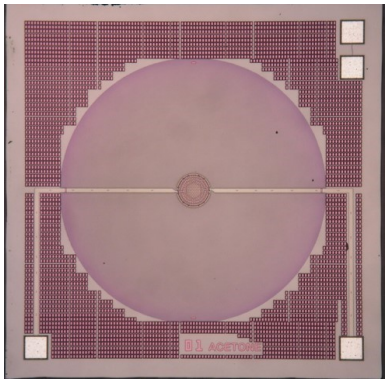

Figure 5: An optical microscopy image of the acetone IR emitter chip (top view, size: 1.6 mm × 1.6 mm).

III. MEASUREMENTS AND RESULTS

These newly designed narrowband plasmonic based IR micro-hotplate sources have been used to replace non-plasmonic emitters, and together with a commercial single channel silicon thermopile detector (HMS-J-21, Heimann Sensor, Germany) formed our novel NDIR sensor testing system. The commercial detectors have optical filters centred at 8.26 µm with a 0.26 µm bandwidth to detect acetone and 10.5 µm with a 0.18 µm bandwidth to detect ammonia. In our setup, the emitter was powered by a current controlled circuit with a fixed current of 16 mA. The detector had an amplified circuit and was connected to the National Instrument data acquisition unit (DAQ) to collect response signals. The gas chamber size for both gases, which is the distance between the emitter and detector, was a 10 cm gold-plated aluminium tube for maximum reflection. The gas inlet was located close to the emitter and the outlet close to the detector. The whole unit was connected to the gas rig, where it was exposed to acetone and ammonia in synthetic air at various concentration and humidity levels. This system enable the detection of acetone in both dry and humid synthetic air at concentrations between 50 ppm and 200 ppm, generated on a gas test bench, with a constant flow rate of 0.5 slpm monitored through mass flow controllers. Low ppm (10 ppm, 15 ppm and 20 pmm) measurements of ammonia in dry and humid air were also performed in the same manner. Each gas concentration was trialled for 2 min, returning to a baseline of synthetic air. To ensure the reliability of our measurements, the experiment was performed with at least three repetitions. Because of significant noise in the signal from the thermopile detector (as shown in Fig. 6), it was first passed through an on-board analogue low pass filter and amplifier. The signal was then sampled, values collected, and post-processed in MATLAB with moving average and median filters to reduce further the noise in the sensor signal. The signal can also be compensated by adding a reference channel to offset changes in the emission of the narrowband IR radiation. The processed data of the sensor response towards acetone pulses in both dry and relative humidity (RH) 25% air are shown in Fig. 6 and Fig. 7, respectively. The experimental results for ammonia detection in

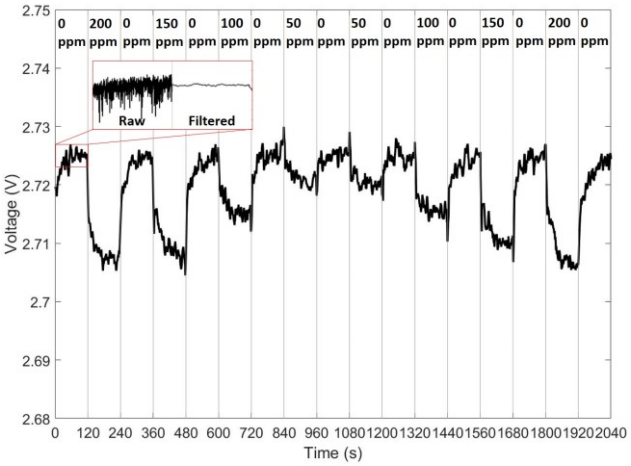

Figure 6: NDIR gas sensor response towards acetone with plasmonic emitter and commercial detector in dry air, from 50 ppm to 200 ppm, in 2-minute steps. (Enlarged view shows raw data versus filtered data.)

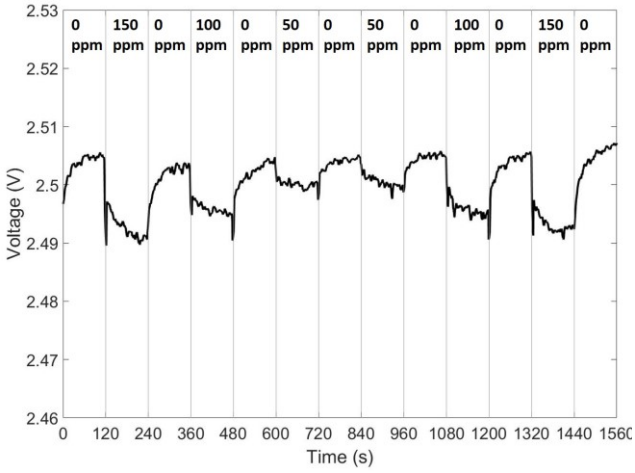

Figure 7: NDIR gas sensor response towards acetone with plasmonic emitter and commercial detector in 25% RH air, from 50 ppm to 150 ppm, in 2-minute steps.

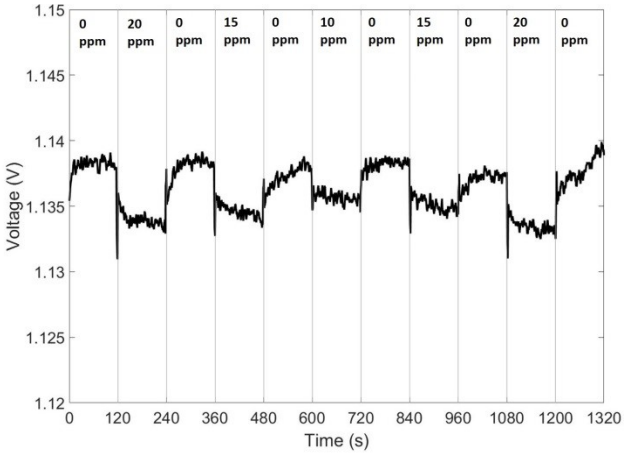

Figure 8: NDIR gas sensor response towards ammonia from 10 to 20 ppm in 2-minute steps, obtained with plasmonic emitter and commercial detector in dry air.

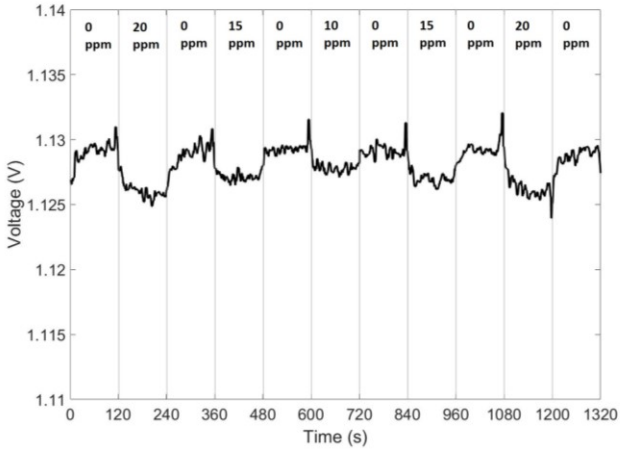

Figure 9: NDIR gas sensor response towards ammonia from 10 to 20 ppm in 2-minute steps, obtained with plasmonic emitter and commercial detector in 25% RH air.

dry conditions and 25% RH air are presented in Fig.8 and Fig. 9. The humid condition lowers the sensors baseline voltage in both gases, and affects their performance by reducing the response by 30%, which can vary depending on the humidity levels. Both sensors responses to acetone and ammonia within 30 ms and their responses increased with the concentration levels.

IV. Development of System for Acetone and Ammonia Monitoring

Based on our designs and testing results, we integrated two NDIR sensors into a portable gas analyser for the detection of target gases. Fig. 10 shows a microcontroller based gas sensor module that can be connected to a laptop or even a smartphone. It is housed inside an aluminium chamber and can be used in harsh conditions such as high temperature, humidity or low oxygen environment. This unit is USB powered and can be adapted for more complex tasks. Apart from the two NDIR sensors, it may also include resistive commercial and laboratory sensors, all assembled in one chamber underneath the NDIR tubes as marked in the figure. This instrument also contains an integrated environmental sensor (Bosch, BME280) for ambient humidity, temperature and pressure monitoring. All sensors are integrated so the unit could be used for comprehensive air quality monitoring, harsh environment exploring, or impurities detection in hydrogen fuel. The unit is prototyped and under further improvement, such as better signal processing capabilities, and new methods for data logging and displaying for easy readout.

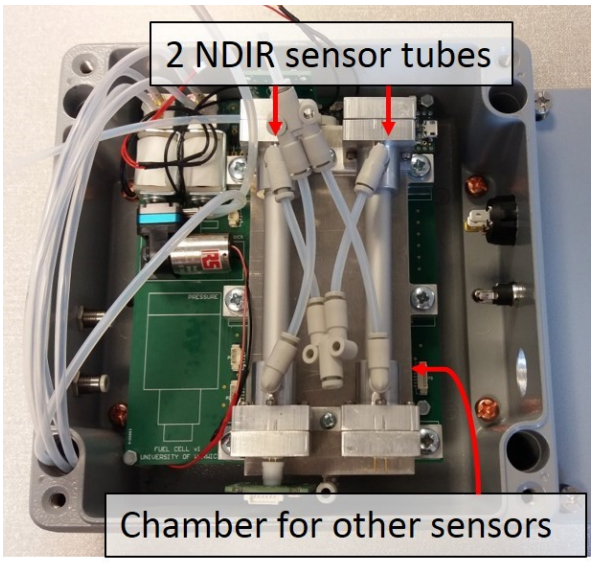

Figure 10: Portable gas sensing system with two integrated NDIR gas sensors (tube length: 10 cm).

V. Conclusions and Futher Works

In this work, a new generation of sensitive low-cost NDIR gas sensors are presented incorporating plasmon-enhanced IR emitters for both acetone and ammonia detection. The emitter design comprised arrays of extruded cylindrical plasmonic structure to increase the infrared radiation only at the target wavelengths of 8.26 µm and 10.6 µm for acetone and ammonia, respectively. These designs were initially modelled and simulated in COMSOL v5.1 with 5% manufacture tolerance and then fabricated in bulk technology. Results from simulations show a close to black body emission at the desired wavelengths and the experimental data correspond well with the results from modelled structures. Such sensors can detect as low as 50 ppm of acetone in both dry and humid synthetic air and 10 ppm of ammonia under same conditions. Our novel NDIR system has been adapted to fit into a portable gas sensing unit which can be used for air quality monitoring, in harsh conditions such as hydrogen environment with high humidity, or low oxygen conditions. The sensitivity can be improved to 1 ppm level by using a dual detector and a sinusoidal emitter signal in order to extract data using Fast Fourier Transform and remove the remaining noise.

Acetone has been investigated in this work as an interesting VOC for air quality monitoring. Ammonia in turn has been of our interest due to its increased toxicity in the environment. Other gases such as carbon monoxide and hydrogen sulphide are also of our interests. They are all currently under development using the NDIR technology based on these novel plasmonic metamaterials.

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