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Characterization of a cMUT as a gas sensor for a fast-response electronic nose

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Abstract—This work presents a characterization of CMUTs as promising candidates for a fast-response, miniaturized electronic nose. A complete setup has been designed, including MEMS chip and packaging, electromechanical valves and dedicated electronics. The measurements focus on the characterization of one channel to demonstrate satisfying sensitivities to some usual volatile organic compounds. The measurements exhibit fast-response to stimuli, highlighting how CMUTs could be a building block of an industrially suitable electronic nose.

Keywords—MEMS, CMUT, gas sensing, gas sensor, electronic nose

I. INTRODUCTION

Relying on hundreds of non-specific receptors, the olfaction is a sense that is yet to be thoroughly reproduced. Digital devices capable of discrimination and recognition between volatile organic compounds (VOCs) are called electronic noses and have been built around the biomimetic idea of arrays of non-specific sensors since the eighties [1]. Nowadays, they answer a real need in environmental challenges, pollution detection, sanitary requirements and comfort needs. They offer a quick and reasonably costly, yet non-specific, alternative between complex lab instruments and simple detectors. The smell, substantially composed of several molecules in suspension in the air, needs to get in contact with a detector to trigger recognition. Thus, an electronic nose is a complex object, capable of chemical exchanges with a gaseous environment, of inhaling i.e. generating a flow, and of generating a signal that will become meaningful when compared to a database and assigned to a smell. Such devices are currently used in perfume, food, or automotive industry [2], [3]. Better performances could widen their field of application to large-scale environmental issues [4] or widespread medical treatments [5], [6]. The aim

is to refine the mimicry of the olfactory sense, as well as better industrialization suitability through miniaturization and cost reduction.

The gravimetric detection abilities of Capacitive Micromachined Ultrasound Transducers (CMUTs) have motivated promising work in the field of detection in gaseous medium [7]–[12]. Their large surface-area-to-volume ratio, combined with low Allan deviation provides them good mass sensitivity and favorable signal-to-noise performances [13]. Furthermore, they offer characteristics of prime interest in the design of an operational electronic nose: electronic-only actuation and readout, which makes an array of several CMUT economical to implement, and a flat surface easy to functionalize.

II. EXPERIMENTAL SETUP

A. CMUT chip

The chip is made of 16 separate elements among which only one is used for the subsequently reported experiments. This element is composed of 1600 individual CMUT membranes, connected in parallel so as to increase the generated signal and the active surface of adsorption (Fig. 1). A single membrane is an 18.5 μm -wide square-shaped stack of silicon (800 nm), SiN (50 nm) and Au (50 nm). The typical resonant frequency of such membranes is 20.8 MHz. The membrane is separated of the bulk by a physical gap (165 nm), 50 nm of SiO₂ and 270 nm of SiN. The wafer are fabricated in LETI's 8" cleanroom using wafer-bonding technology and SOI wafer. The fabrication process can be found in [14]. The silicon top layer is used as the top electrode while the bulk silicon is used as the bottom, ground electrode. A DC polarization bias of 25 V as well as an AC actuation signal of 1 V are applied to the top electrode. The connection between the chip and the electronics is made by wire-bonding the chip to a PCB that can be plugged to the motherboard described later. On top of the surface, a fluidic chamber is created by a hood containing the chip and screwed to the PCB.

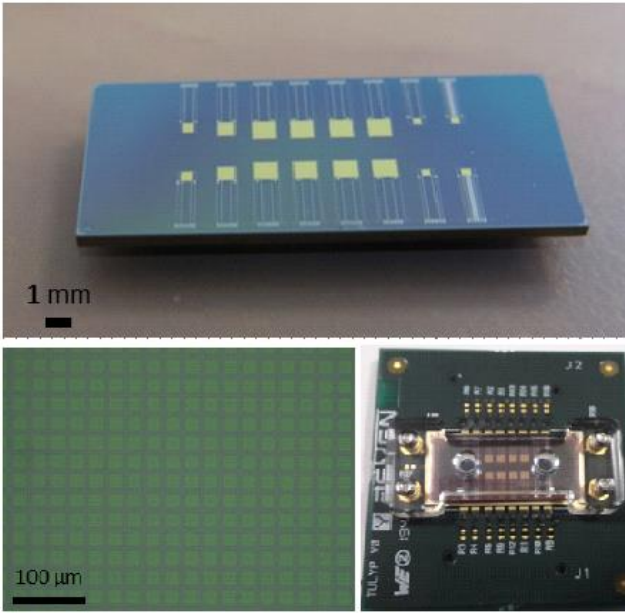


Fig 1: CMUT chip with elements made of 1600 (used), 900, 400 and 196 (not used) individual CMUTs membranes (top), array of CMUTs membranes (bottom left), PCB with the hood containing the chip (bottom right).

B. Electronics

The electronics has been developed to actuate and read out the sensors to track with precision the variations of the resonant frequency of the CMUTs. The schematics of the circuit, based on a phase lock loop, is shown in Fig. 2. The CMUT is actuated by a drive signal close to the resonant frequency.

A lock-in demodulator is used to determine the phase of the signal arising from the CMUT. A phase controller feedbacks the drive frequency so that the error phase is maintained close to zero. Therefore, the drive frequency follow the resonant frequency, which vary during an experiment when additional mass is adsorbed.

A more simple, “open loop” mode, may also be used. In this case, the drive frequency is set by the user and the electronics delivers the current phase as an output, which is proportional to the frequency shift by a factor $-f/2Q$, with f the nominal frequency and Q the quality factor [15].

In both cases, the tracking of such precise variations, as low as a few Hz over a 20 MHz nominal frequency, is allowed by a compensation of the nominal capacitor of the CMUT. The drive excitation actuates an external capacitance, with a π

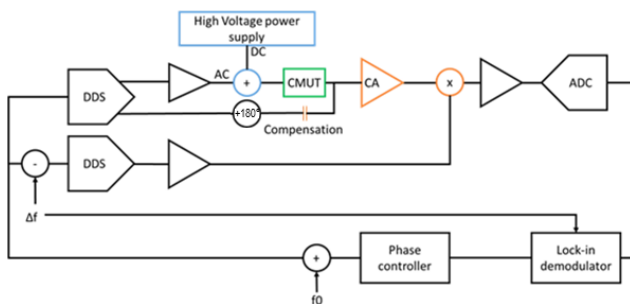


Fig. 2: Phase lock loop actuation and readout electronics schematics.

radian phase shift, which generates a current that is added to the CMUT’s current. The resulting current is more appropriate to reveal the mechanical behavior of the membranes, otherwise somehow diluted in the capacitance of the chip.

C. Fluidics

A fluidic line allows controlled injections of VOCs diluted in the ambient air. Ambient humid air is used to reproduce the complex and dynamic conditions of the real situations where an electronic nose is expected to be used. The VOC is pumped from the headspace of a bottle, through dilution and electromechanical valves, to fill the fluidic chamber inside which the flow will interact with the surface of the CMUT chip. The flow then goes to the exhaust through a set of characterization sensors such as a photoionization detector to measure *in situ* the concentration of VOC.

D. Measurement protocol

A typical measurement guided by electronic nose’s requirements ([16], [17]) consists of tracking the signal of interest, in our case the frequency, during three steps: the baseline, or exposition to ambient air during a few seconds, the injection phase, with the compound of interest in the flow, and the desorption phase with recovering ambient air to renew the surface for the following measurement (Fig. 3). The amplitude of the signal is the frequency shift between the baseline and the plateau reached after a few seconds of injection. Fluidics paths have been carefully compensated to avoid any impact of head losses on the frequency during injections. The flow is set to 50 mL/min, sufficiently low to consider the gas in the headspace at saturation vapor pressure. For repeated measurements, the sensor is exposed to ambient air during 40 seconds between 20 seconds injections of the VOC of interest.

III. RESULTS

A. Noise floor

As preliminary measurements, the noise floor has been measured in both open and close loop. The two modes are equivalent with a multiplication factor of 125 000, which

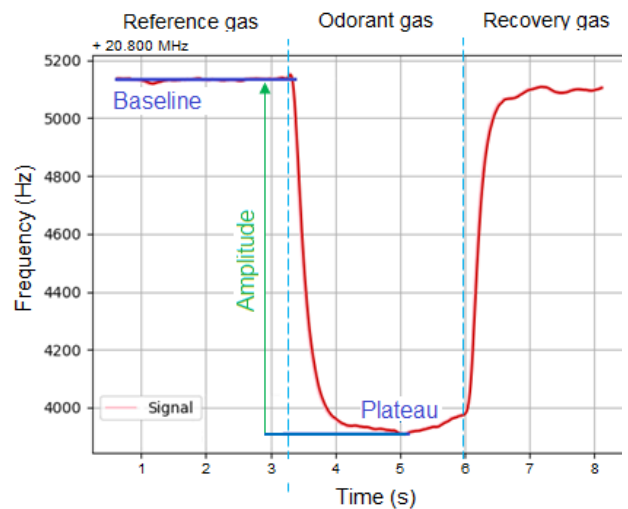


Fig. 3: Three-steps measurement and extraction of the value of interest.

corresponds to a quality factor of 80 experimentally observed on impedance measurements (not shown). Open-loop and closed-loop Allan deviation shows the same minimum value of $3 \cdot 10^{-7}$ at 1s (Fig. 4). At short integration times, the open-loop measurements reveal a slope corresponding to frequency white noise [16], attributed to the first amplifier stage, while the response of the closed loop signal is limited by the controllers' time response hence does not reflect the CMUT's behavior.

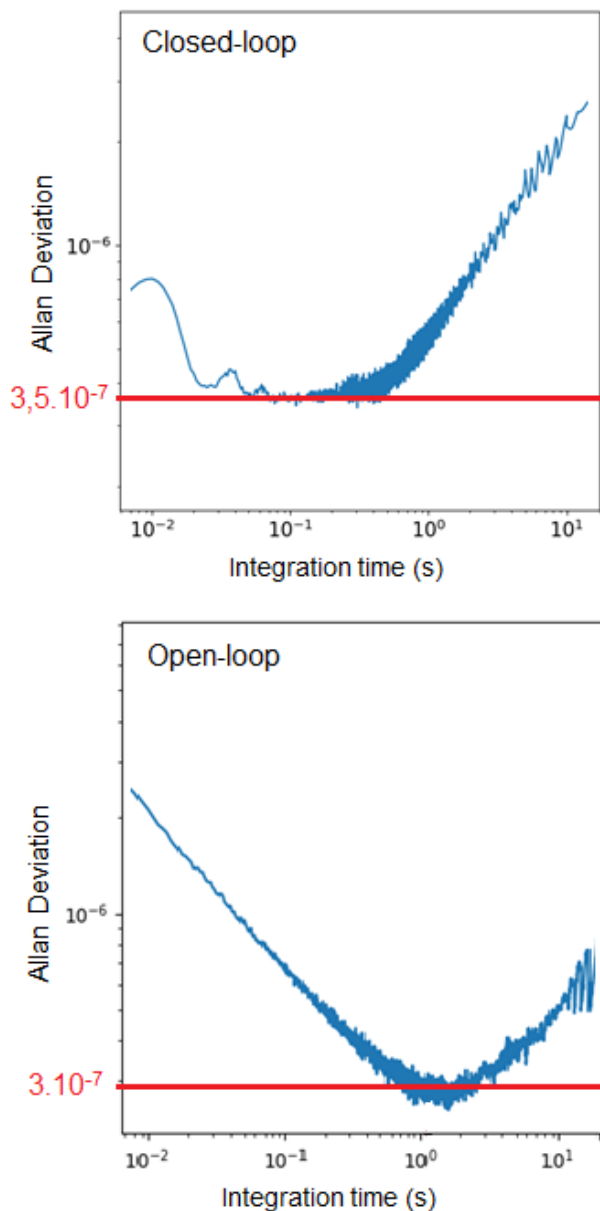


Fig 4 : Allan deviation in closed loop (top) and open-loop (bottom) showing a minimum value of $3 \cdot 10^{-7}$ i.e. 6Hz. The multiplication factor applied to open loop measurement is $-125 \cdot 10^3$, corresponding to a quality factor of 80.

B. Sensitivity

Three compounds are used in the following experiments: butanol, a fermentation alcohol present in wines and food, nonane, a low flammability hydrocarbon present in gas-oil, and beta-pinène, emitting a characteristic smell of forest. Each of these VOCs are measured for a wide range of

concentration, and then a sensitivity is estimated from the signals (Fig. 5). The sensitivity of the sensor has been found to be 0.05 Hz/ppm, 0.12 Hz/ppm and 0.42 Hz/ppm, respectively for beta-pinène, nonane, and butanol. Considering a minimal measurable signal of 20 Hz (3σ), the limit of detection are 400 ppm, 170 ppm, and 50 ppm, respectively for beta-pinène, nonane, and butanol.

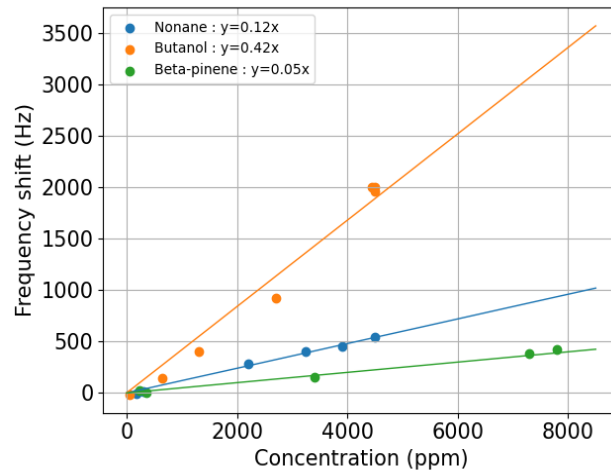


Fig. 5 : Frequency shift vs VOC concentration for nonane (blue), butanol (orange) and beta-pinène (green). Sensitivity is extracted by a linear fit.

IV. DISCUSSION

The chemical sensitivity relies on the mass sensitivity of the gravimetric sensor, on the molar mass of the compound, and on its tendency to adsorption. The mass sensitivity of the sensor is identical for all experiments and estimated at 10 Hz/fg. Experiment shows how adsorption plays a crucial role, as the sensitivities for each compound are arranged in descending order of molar mass, but in ascending order of vapor saturation pressure (i.e. adsorption tendency) as listed in Table 1. Measurements with octanol (molar mass of 130 g/mol; vapor saturating pressure of 0.003 kPa at 20°C) reveal a sensitivity above 10 Hz/ppm (not shown).

TABLE I. VOCs SENSITIVITIES AND CHEMICAL CHARACTERISTICS

Compound	Sensitivity (Hz/ppm)	Molar mass (g/mol)	Vapor saturation pressure at 20°C (kPa)
Butanol	0.42	76	0.93
Nonane	0.12	128	0.71
Beta-pinène	0.05	136	0.39

The time of response of the system presented in this work is typically around one second, which is quicker by two or three orders of magnitude compared to literature on electronic noses using CMUTs. The desorption phase also shows a quick return to the baseline frequency, highlighting complete renewal of the surface. Usual functionalization uses polymer coatings, relying not only on adsorption but also on absorption of the VOC by the material. This can lead to higher sensitivities but nonetheless lengthens the characteristic response time of the sensor, which is a key

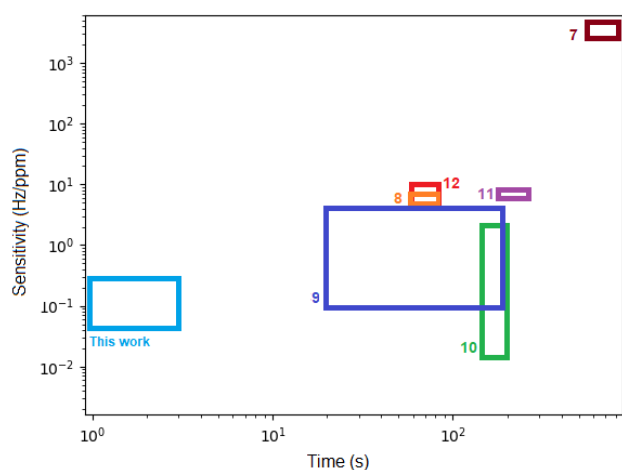


Fig. 6 : Comparison between ranges of sensitivity and typical response time between CMUT electronic noses in the literature. Response time relates to the time to reach 90% of the intensity of the signal, extracted from references [7]–[12].

parameter of an electronic nose. Among others, Stedman et al. [9] reported its best sensitivity of 8 Hz/ppm with methanol on P4VP as a polymer coating for a time response of 30 seconds. Fig. 6 merges the response time and sensitivity of some works ([7]–[12]), highlighting how the relatively low sensitivity reported in this work is balanced by the fast response time.

Peptide-based functionalization combined with Surface Plasmon Resonance Imagery have already demonstrated ability to discriminate VOCs [19] with a fast response time [20]. Additional experiments (not shown) carried out on CMUT surfaces covered by similar peptide-based coating have confirmed that this coating do not induce noticeable increase in response time, which open promising research tracks for fast-response, peptide-based-coated electronic noses based on CMUT technology.

V. CONCLUSION

We implemented a CMUT chip in an experimental setup, consisting of dedicated electronics able to actuate and readout a CMUT element, and a fluidic line allowing precise and calibrated injections of VOCs. The system demonstrates an Allan deviation of 3.10^{-7} or 6 Hz at 1s. One CMUT element was exposed to different VOCs. Sensitivities up to 0.42 Hz/ppm have been measured with relatively highly volatile compounds, with a response time as low as one second. Future work will consist of simultaneous measurements of an array of differently peptide-based functionalized CMUTs in order to show the ability of the system to discriminate VOCs.

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