Proof of Concept and Preliminary Results of Gas Detection by Measuring the Admittance at the Resonance and Anti-resonance of an Uncoated CMUT

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Functionalized films are commonly used in gas sensing to target a particular gas. This is due, in part, to their usually high capability to detect very low concentrations and their high selectivity. However, their poor stability over time remains a challenge when dealing with applications that require the sensing to remain reliable without frequent recalibration. For this reason, uncoated gas sensors have become increasingly popular regardless of their lower sensitivity and their often non-selective characteristics. There exist different approaches for gas sensing without a functionalized film. One possibility is to use an uncoated resonating sensor and tracking its resonant properties which depend on its surrounding environment. The easy integration capability of capacitive micromachined ultrasonic transducers (CMUTs) makes them great candidates for uncoated gas sensing. Moreover, they are able to reach very high resonant frequencies and, therefore, allow for a shorter response time. In this article, a method to detect gas by following the value of the admittance of an uncoated silicon nitride CMUT array at either the resonant or at the anti-resonant frequency is presented and tested. This chemical detection is purely based on the change of the physical properties of the gas mixture (the mass density and the viscosity). A general model describing the impact of the electrical and mechanical properties of the CMUT in the sensitivity is presented, validated and applied to carbon dioxide and methane detection.

Keywords: CMUT, capacitive sensor, MEMS, uncoated sensors, gas sensing, resonator

1. INTRODUCTION

In gas sensing, coating a device with a thin film specifically tailored to chemically interact with the target analyte is by far the most common approach (Göpel, 1994; Park et al., 2007; Lee et al., 2011; Qiu et al., 2015). However, it has been shown that using a sensitive coating compromises the long term stability of the sensor (Korotcenkov and Cho, 2011) mostly due to aging of such film (Sharma et al., 2001; Romain and Nicolas, 2010). For this reason, there is an increasing interest toward uncoated sensors, which rely on the change of the physical properties of their surrounding gas such as thermal conductivity (Daynes, 1933; Hale et al., 1992; Simon and Arndt, 2002), or mass...
density (Tétin et al., 2010; Boudjiet et al., 2014; Rosario and Mutharasan, 2014). Despite the lack of selectivity of uncoated sensors, it is still possible to discriminate from different gases by measuring multiple parameters at once (Iglesias et al., 2019).

It has been shown that both the resonant frequency and the quality factor of uncoated resonant sensors such as microcantilever resonators depend on physical properties of the fluid surrounding them (Sader, 1998; Abdolrazzaghi and Daneshmand, 2016). This has been used previously for characterizing liquids (Heinisch et al., 2015) and even gases, despite the fact that their mass density and viscosity are much smaller. Gas detection such as hydrogen, carbon dioxide, argon, helium and methane detection have already been demonstrated using uncoated resonant sensors (Tétin et al., 2010; Boudjiet et al., 2014; Rosario and Mutharasan, 2014; Iglesias et al., 2019). These measurements have been performed either using silicon or piezoelectric cantilevers at frequencies below 200 kHz. However, capacitive micromachined ultrasonic transducers (CMUTs) (Haller and Khuri-Yakub, 1996) present an advantage over piezoelectric resonators due to their relatively easy integration (Törndahl et al., 2002; Wygant et al., 2008; Gurun et al., 2011; Um et al., 2014). Additionally, gas sensing has already been done with uncoated ultrasonic microresonators by measuring the time of flight of a signal propagating through the gas (Toda and Kobayakawa, 2008; Shanmugam et al., 2018).

Nevertheless, a physical gas detection using the measurement of the admittance at the resonance and anti-resonance and not on the corresponding frequency shifts, along with an analytical model of the impact of a change in the gas composition, are presented and verified under mixtures of nitrogen and either carbon dioxide (CO₂) or methane (CH₄) as an example.

The principle of this method along with the experimental setup and results will be presented in the following sections.

2. MATERIALS AND METHODS

2.1. Sensor and Model

Schematics of a single Capacitive Micromachined Ultrasonic Transducer (CMUT) like the one used in this study are shown in Figure 1A. The microfabrication process based on surface micromachining can be found in Heller et al. (2018). By applying a voltage \( V \) between the electrodes, the silicon nitride membrane deflects, changing the capacitance of the sensor.

It has been shown previously that a simple lumped model as the one in Figure 1B can describe accurately the behavior of a single CMUT (Wenchao et al., 2008; Koymen et al., 2017) and it results in the following equation corresponding to Newton’s second law of motion:

\[
\frac{\partial^2 u}{\partial t^2} = \frac{1}{2} \frac{\epsilon_0 A V^2}{(h_{eq} - u)^2} - c \frac{\partial u}{\partial t} - ku,
\]

where \( k \) is the stiffness of the membrane, \( c \) the linear damping coefficient (depending on the viscous losses in the gas), \( m \) the displaced mass (of both the membrane and the surrounding gas), \( u \) the displacement of the membrane, \( V \) the actuation voltage, \( \epsilon_0 \) the vacuum dielectric constant, \( A \) the surface of the membrane and \( h_{eq} \) the equivalent gap of the CMUT, whose expression is:

\[
h_{eq} = h_{gap} + \frac{t_m}{\epsilon_r},
\]

where \( h_{gap} \) is the gap between the dielectric membrane and the bottom electrode at rest (\( V = 0 \)), \( t_m \) the thickness of the membrane and \( \epsilon_r \) the relative permittivity of the membrane.

In such case, by considering that:

\[
V = V_{dc} + V_{ac},
\]

where \( V_{dc} \) is the bias and \( V_{ac} \) the excitation voltage, it can be shown that, at a given \( V_{dc} \) such that \( V_{ac} << V_{dc} \), the harmonic electrical admittance \( Y \) of a single CMUT can be expressed as:

\[
Y = j \omega C_0 \left( \frac{1 + j^2 \omega_k}{k_1} \right) \left( 1 + \frac{V_{dc}^2}{2 k_1^2 \omega_k^2} \right),
\]

where \( j \) is such that \( j^2 = -1 \), \( \omega \) the angular frequency of the excitation, \( C_0 \) the static electrical capacitance and \( k_1 \) the stiffness of the membrane by taking into account the electrostatic softening. The expression of \( k_1 \) and \( C_0 \) are:

\[
k_1 = k - \frac{\epsilon_0 A V_{dc}^2}{(h_{eq} - u_{dc})^2},
\]

\[
C_0 = \frac{\epsilon_0 A}{h_{eq} - u_{dc}},
\]

where \( u_{dc} \) is the static displacement of the membrane corresponding to \( V = V_{dc} \).

From Equation (4), under vacuum and assuming no solid viscous losses (Koymen et al., 2017) (\( c = 0 \)), the vacuum admittance resonant (\( \omega_{r,0} \)) and anti-resonant (\( \omega_{a,0} \)) angular frequencies can be defined. Their expressions are:

\[
\omega_{r,0} = \sqrt{\frac{k_1}{m}},
\]

\[
\omega_{a,0} = \sqrt{\frac{k}{m}}.
\]

Similarly, the corresponding quality factors (\( c \neq 0 \)) of the admittance resonance (\( Q_r \)) and anti-resonance (\( Q_a \)) can be defined:

\[
Q_r = \frac{\sqrt{k_1 m}}{c},
\]

\[
Q_a = \frac{\sqrt{k m}}{c}.
\]

The CMUT’s electromechanical coupling coefficient (\( k_t \)) (Yaralioglu et al., 2003) is introduced as:

\[
k_t^2 = 1 - \left( \frac{\omega_{r,0}}{\omega_{a,0}} \right)^2 = \frac{2 u_{dc}}{h_{eq} - u_{dc}}.
\]
Equations (6), (7), and (9) can then be rearranged:

\[
C_0 = \frac{\varepsilon_0 A (2 + k_t^2)}{2 h_{eq}},
\]

(12)

\[
\omega_{r,0} = \omega_{a,0}\sqrt{1 - k_t^2},
\]

(13)

\[
Q_r = Q_a\sqrt{1 - k_t^2},
\]

(14)

which leads to the final expression of \( Y \):

\[
Y = j\omega C_{00} Y_n,
\]

(15)

where \( C_{00} \) is the capacitance without bias \( (k_t = 0) \):

\[
C_{00} = \frac{\varepsilon_0 A}{h_{eq}},
\]

(16)

and \( Y_n \) the normalized admittance containing all the information about the resonant properties of the CMUT. Its expression is:

\[
Y_n = \frac{(2 + k_t^2)}{2(1 - k_t^2)} \frac{1 + j\frac{1}{Q_a(1-k_t)} x - x^2}{1 + j\frac{1}{Q_a(1-k_t)} x - x^2},
\]

(17)

where:

\[
x = \frac{\omega}{\omega_{a,0}}.
\]

(18)

The modulus of the reduced admittance \(|Y_n|\) is maximum at \( x = x_r \) and minimum at \( x = x_a \):

\[
x_r = \sqrt{1 - \frac{k_t^2}{2}} - \frac{\sqrt{Q_a^2 k_t^4 - 2k_t^2 + 4}}{2Q_a},
\]

(19)

\[
x_a = \sqrt{1 - \frac{k_t^2}{2}} + \frac{\sqrt{Q_a^2 k_t^4 - 2k_t^2 + 4}}{2Q_a},
\]

(20)

Their corresponding values, respectively, \(|Y_n|_{\text{max}}\) and \(|Y_n|_{\text{min}}\), are:

\[
|Y_n|_{\text{max}} = \frac{2 + k_t^2}{2} \frac{\sqrt{Q_a^2 k_t^4 + 4x_a^2 + \delta(1 + Q_a^2 k_t^2)}}{\sqrt{Q_a^2 k_t^4 + 4k_t^2 + \delta(1 - Q_a^2 k_t^2)}},
\]

(21)

\[
|Y_n|_{\text{min}} = \frac{2 + k_t^2}{2} \frac{\sqrt{Q_a^2 k_t^4 + 4x_a^2 - \delta(1 + Q_a^2 k_t^2)}}{\sqrt{Q_a^2 k_t^4 + 4k_t^2 - \delta(1 - Q_a^2 k_t^2)}},
\]

(22)

where:

\[
\delta = x_a^2 - x_r^2 = \frac{1}{Q_a^2} \sqrt{Q_a^2 k_t^4 - 2k_t^2 + 4}.
\]

(23)

Therefore both, \(|Y_n|_{\text{max}}\) and \(|Y_n|_{\text{min}}\), depend only on both \( Q_a \) and \( k_t \). From Equation (11), one could argue that, at a fixed polarization, \( k_t \) still depends on the gas through \( \omega_{r,0} \) or \( \omega_{a,0} \). However, from Equation (1) in static:

\[
0 = \frac{1}{2} \frac{\varepsilon_0 AV_d^2}{h_{eq} - u_d} - k u_d,
\]

(24)

\( u_d \) depends only on \( k, \varepsilon_0, A, V_{dc}, \) and \( h_{eq} \). None of which depend on the gas properties. This means, according to Equation (11), that at fixed polarization \( k_t \) is constant. However, a change in the gas composition induces a change \( dQ_a \) in \( Q_a \). Therefore one can expect to see a change in \(|Y_n|_{\text{max}}\) and \(|Y_n|_{\text{min}}\) as well. At this point, it should be highlighted that \( x_r \) is only defined for values of \( k_t \) below a critical value \( k_c \) for which \( x_r = 0 \):

\[
k_c = \sqrt{2 - \frac{2Q_a^2}{2Q_a^2 - 1}} \approx 1,
\]

(25)

which happens close to the collapse voltage \( V_{\text{coll}} \). Finally, by differentiating Equations (21) and (22), at fixed \( k_t \), it comes:

\[
\frac{d|Y_n|_{\text{max}}}{|Y_n|_{\text{max}}} = \frac{\alpha_r}{Q_a} \frac{dQ_a}{Q_a},
\]

(26)

\[
\frac{d|Y_n|_{\text{min}}}{|Y_n|_{\text{min}}} = \frac{\alpha_a}{Q_a} \frac{dQ_a}{Q_a},
\]

(27)
where $\alpha_r$ and $\alpha_a$ are the electrical gains in sensitivity at the resonance and anti-resonance, respectively. Their expressions are:

$$
\alpha_r = \frac{-4k_r^2x_r^2Q_r^2}{8x_r^2 + 2Q_r^2k_r^2 + \delta[3 + 2Q_r^2(k_r^2 - 2)]},
$$
$$
\alpha_a = \frac{-4k_a^2x_a^2Q_a^2}{8x_a^2 + 2Q_a^2k_a^2 - \delta[3 + 2Q_a^2(k_a^2 - 2)]}.
$$

By analyzing the difference $\Delta \alpha$:

$$
\Delta \alpha = |\alpha_r| - |\alpha_a| = \frac{4Qa^2k_t^2}{(2Qa - 1)(2Qa + 1)[4Qa^2(k_t^2 - 1) + 1]},
$$

it is possible to show that $\Delta \alpha < 0$ for $k_t^2 < 1 - 1/(4Q_a^2)$ which is the case in practice since $k_t^2 < 1 - 1/(4Q_a^2)$. Therefore, the sensitivity is always higher by tracking $|Y_n|_{min}$ instead of $|Y_n|_{max}$. That being said, for typical values of $Q_a$ (10–1,000) and $k_t^2$ (below 0.3), this difference is rather small (below 1%).

It is worth noticing that the formulae of this section 2.1 are the same for an array of identical CMUTs in parallel like the one shown in Figure 2. Nevertheless the overall variations are added with a higher number of CMUTs, which improves the signal to noise ratio.

At this point, the model can be applied to all kinds of sensing where the principle is to track the admittance peaks of a CMUT. In the particular case of gas sensing with an uncoated CMUT array, $m$ and $c$ will change due to the effect of the gas (air or N$_2$ for instance) and will modify the quality factor $Q_d$. Finally, the sensitivity to a particular gas will depend both on its viscosity and its mass density through $dQ_a$ (viscous losses).

### 2.2. Model Validation

In order to compare the model, some parameters of the model need to be determined by the measured admittance $|Y(\omega)|$.

The parameters $\omega_r,0$ (in N$_2$) and $Q_r$ (in N$_2$) were extracted from the measured conductance $G$, which is the real part of the admittance $Y$:

$$
G = \omega C_0 \frac{k_t^2}{1 - k_t^2} \frac{\omega}{\omega + \omega_r,0Q_r} \left( \frac{\omega}{\omega + Q_0Q_r} \right)^2,
$$

(31)

Indeed, the maximum of $G$, named $G_{max}$, is for $\omega = \omega_r,0$ and $Q_r$ can be estimated by using:

$$
Q_r = \frac{\omega_r,0}{\Delta \omega},
$$

(32)

where $\Delta \omega$ is the bandwidth at $G = G_{max}/2$.

$C_0$ was obtained by measuring the slope of $|Y(\omega)|$ at $V_{dc} = 0$V as shown in Figure 3A for which the value $C_0 = 190\mu F$ was obtained. Knowing the value of $C_0$ allowed to compute $|Y_n|_{n} = |Y|/|C_0(\omega)|$ for different values of $V_{dc}$. It allowed to determine the value of $k_t$ by using Equation (33):

$$
k_t^2 = \frac{2H_0 - 2}{2H_0 + 1},
$$

(33)

where:

$$
H_0 = \lim_{x \to 0} |Y_n|(x) = \frac{2 + k_t^2}{2(1 - k_t^2)}.
$$

(34)

Knowing $\omega_r,0$, $Q_r$ and $k_t$, $\omega_a,0$ = 50 rad/$\mu$s and $Q_a$ = 33 are simply obtained by using Equations (13) and (14), respectively.

Figure 3B shows the experimental values of $|Y_n|$ for $V_{dc}$ = 15V and its expression (Equation 17) resulting from the lumped model approximation (dashed) as a function of $\omega$. Both curves are in agreement with each other. The mismatch can be explained by the simplicity of the model. Indeed, neither a particular geometry nor a non-uniformity between the array elements (Lee et al., 2009) have been considered. Also, in order to verify experimentally that the sensitivity at the resonance is comparable at the one at the anti-resonance, the relative
change in admittance was measured in both cases while the gas composition was changed from pure N$_2$ to 80% of CO$_2$ in N$_2$. The results, shown in Figure 4, confirm the barely higher sensitivity in the case of the anti-resonance. Moreover, in Figure 4, the relative change of the resonant angular frequency, $\alpha_n$, during the same experiment has been plotted. It can be easily concluded that, with this particular device, tracking the admittance at either the resonance or anti-resonance is much more sensitive than the typical method of tracking the resonant angular frequency (gas detection step only visible in the inset of Figure 4).

Additionally, verification of the expression of $\alpha_a$ from Equation (29) is shown in Figure 5 where the theoretical values are shown for different values of $Q_a$ as a function of $k_t$ (solid lines). The experimental values were obtained through Equation (27) by monitoring $|Y_n|_{\text{min}}$ for $V_{dc}$ varying from 1V to 52V (90% of $V_{coll}$) in order to avoid damaging the sensors. For each value of $V_{dc}$, 4% of CO$_2$ was introduced in order to be able to measure $d|Y_n|_{\text{min}}$ even for low values of $k_t$. The obtained $dQ_a$ from the obtained values of $Q_a$ is $dQ_a = -0.126$ (for the case $V_{dc} = 35V$). These values are also plotted (orange dots) and are in agreement with the theory. The obtained values are reported in Table 1.

Intuitively (and this is confirmed by the plot of $\alpha_a$ in Figure 5), for gas detection, one should try to get as close to $V_{coll}$ as possible (taking into account $V_{dc}$) in order to reach the highest sensitivity. However, the noise standard deviation ($\sigma$) increases as well with $V_{dc}$ (Wygant et al., 2016) and, therefore, the limit of detection (LOD), which can be approximated by Equation (35) (Lange et al., 2002), does not necessarily improve.

$$LOD = \frac{3\sigma}{S},$$

where $S$ is the overall sensitivity to a particular gas. For this reason, the results presented in the following section were carried out at 60% of $V_{coll}$ ($V_{dc} = 35V$) where the best trade-off between noise and sensitivity was measured.

### 3. RESULTS AND DISCUSSION

#### 3.1. Changing the Gas

In order to test the performances of this method, tests where pure N$_2$ is alternated with either CO$_2$ (heavier gas) or CH$_4$ (lighter gas) at different concentrations (4%, 3%, 2%, 1%) were performed. The relative variation of $|Y_n|_{\text{min}}$ with respect to N$_2$ ($d|Y_n|_{\text{min}}/|Y_n|_{\text{min}}$) for both sequences is shown in Figures 6A,B, respectively. The temperature drift was compensated by removing the slow varying background.

These results where used afterwards to plot the calibration curves (Figure 7) which show good linearity (at least for that concentration range). Therefore, the sensitivity of the sensor $S$ can be defined for each mixture (N$_2$ and either CO$_2$ or CH$_4$) as the slope of the calibration curve. These values are $S_{CO2} = 6.9 \times 10^{-4}/\%$ and $S_{CH4} = -5.3 \times 10^{-3}/\%$.

In the simpler case of a cantilever respecting Euler-Bernoulli conditions and for small variations, $d\rho$ and $d\eta$ of the mass density $\rho$ and the viscosity $\eta$ of the gas, it can be shown that the quality factor $Q$ varies (Iglesias et al., 2019):

$$\frac{dQ}{Q} = -\frac{a}{\rho} \frac{d\rho}{\rho} + b \frac{d\eta}{\eta},$$

where $a$ and $b$ are positive constants depending on the geometry and the main gas (N$_2$ in this case). Reported values of $d\rho/\rho$ and $d\eta/\eta$ for 1% of either CO$_2$ and CH$_4$ are presented in Table 2.

Therefore, since for CH$_4$ $d\rho$ and $d\eta$ (with respect to N$_2$) are both negative, the sign of $S_{CH4}$ is consistent with the theory ($\alpha_a < 0$). Moreover, given the sign of $S_{CO2}$ and considering that, in the case of CO$_2$, $d\rho > 0$ and $d\eta < 0$ this indicates that the effect of the mass density is dominant over the one of the viscosity. Indeed, by applying Equation (36) to CO$_2$ and CH$_4$ to
TABLE 1 | Experimental values used in Figure 3.

| $V_{dc}$ (V) | 1   | 15  | 30  | 40  | 46  | 50  | 52  |
|------------|-----|-----|-----|-----|-----|-----|-----|
| $k^2$ (%)  | 0.2 | 0.9 | 4   | 7   | 11  | 17  | 21  |
| $d[Y_{n\text{min}}/Y_{n\text{min}}]$ (%) | 0.15 | 1.0 | 2.0 | 2.9 | 3.0 | 3.3 | 3.5 |

FIGURE 6 | Measurement for $V_{dc} = 35V$ of $d[Y_{n\text{min}}/Y_{n\text{min}}]$ (drift removed) by alternating pure $N_2$ and either $CO_2$ (A) or $CH_4$ (B) in different concentrations (4%, 3%, 2%, 1%, and 4%).

TABLE 2 | Measured values of $S$ for $V_{dc} = 35V$ and reported values of $d\rho/\rho$ and $d\eta/\eta$ according to previous work (Iglesias et al., 2019).

| Mixture | $N_2$-$CO_2$ | $N_2$-$CH_4$ |
|---------|--------------|--------------|
| $S$ (%) | $6.9.10^{-4}$ | $-5.3.10^{-4}$ |
| $d\rho/\rho$ (%) | 0.56 | -0.42 |
| at 1% in $N_2$ and 20°C 1atm | |
| $d\eta/\eta$ (%) | -0.20 | -0.41 |
| at 1% in $N_2$ and 20°C 1atm | |

$Q_a$ with values from Table 2, it can be shown that $|a/b| = 5.6$. This means that (assuming that Equation 36 holds for a square membrane) this sensing method would be more suitable to detect gases that would induce an important change in the gas mass density $\rho$ rather than for gases that would only induce it in the gas viscosity $\eta$. That being said, the influence of the viscosity remains non-negligible.

3.2. Limit of Detection

Once the sensitivity measured, an estimation of the limit of detection (LOD) for each gas ($CO_2$ or $CH_4$) can be estimated (Equation 35). For this, the noise standard deviation of $d[Y_{n\text{min}}/Y_{n\text{min}}]$ ($\sigma = 0.11\text{permil}$) was measured under pure $N_2$. The computed values both for $CO_2$ and $CH_4$ (respectively $LOD_{CO2} = 0.5\%$ and $LOD_{CH4} = 0.6\%$) were then tested experimentally with an appropriate sequence of lower concentrations for each gas (1%, 0.75%, 0.5%, 0.25%, 1% for $CO_2$ and 1%, 0.8%, 0.6%, 0.4%, 1% for $CH_4$). The results are shown in Figure 8 and are consistent with the calculations.

4. CONCLUSION

In conclusion, an analytical model of the influence of the bias voltage in the sensitivity when measuring the value of the normalized admittance at either the resonance or the anti-resonance of a CMUT was presented. This model showed that the sensitivities at both the resonance and anti-resonance are comparable although slightly higher at the anti-resonance. Experiments have been performed by measuring the admittance value at the anti-resonance which is much more sensitive for the tested device than measuring the resonant frequency which is more classical in the community of resonant gas sensors. The expression of the sensitivity at the anti-resonance was verified at different values of the bias voltage in the context of gas sensing without sensitive coating. Examples of measurements were presented for mixtures of $N_2$ and either $CO_2$ or $CH_4$ for which a sensitivity of $6.9.10^{-4}\%$ and $-5.3.10^{-4}/\%$ were
obtained, respectively, and limits of detection of 0.5\% and 0.6\% were demonstrated, respectively. Such values could potentially be improved by further optimization of other parameters such as the geometry of the CMUT. Combining the presented measurements with other sensors could also compensate for the lack of selectivity to extend the capabilities beyond binary mixtures.

**DATA AVAILABILITY STATEMENT**

The datasets generated for this study are available on request to the corresponding author.

**AUTHOR CONTRIBUTIONS**

PS, J-FM, DA, and DC have designed and fabricated the CMUTs. LI and ID have developed the modeling and data treatments. LI has done the measurements. All the authors have contributed to the writing of the paper.

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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