Examples of vibrating MEMS sensing physical parameters for chemical gas detection

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Abstract: This chapter presents advantages and drawbacks to achieve chemical detection without the use of sensitive coating by measuring physical properties of gas. Example of such detections are presented using two kinds of vibrating MEMS: microcantilevers and capacitive micromachined ultrasonic transducers (CMUT). The main advantages of such sensors relying on their concept are their short response time, good reliability and no need for calibration during the sensor's life cycle. Their major drawback is that there is no selectivity if only one physical parameter measurement is done. To overcome this drawback, discrimination between different gas mixtures can been achieved by simultaneous measurements of multiple physical properties of the gas. This latter principle has been applied with either microcantilevers or CMUT with the simultaneous measurements of either viscosity and mass density or acoustic wave speed and attenuation of gas.

Keywords: Resonant MEMS, microcantilever, CMUT, gas detection, viscosity, mass density, sound speed, time of flight, sound absorption

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1. Context - general principle

Chemical microsensors have not had the same industrial development as physical microsensors, such as accelerometers or gyroscopes for example [1]. There are many reasons for this, including the facts that:

- the markets are niche markets specific to the species to be detected (*bottleneck a*),
- for each of the species to be detected, an appropriate sensitive layer must be developed or chosen (*bottleneck b*),
- chemical sensors are less reliable and have shorter life spans than physical sensors and they have greater drift (*bottleneck c*).

Classically, a chemical microsensor is composed of a transducer and a sensitive layer. The role of the sensitive coating is to interact with the targeted gas. This interaction causes changes in temperature, refractive index, electrical properties, mass or mechanical changes and the transducer transforms these changes into an electrical signal. The sensitive coatings are subjected to environmental effects including temperature, humidity and aging effects. Then, the long-term stability of the coatings and the resulting aging affect the reliability of the sensor more than the aging of the transducer itself.

An alternative method to detect and quantify chemical species is to use "physical sensors" which measure a physical property of the gas without need of chemical interaction with the sensor. Examples of physical properties of gas that can be measured are mass density, viscosity, thermal conductivity and sound speed. These physical properties for major industrial gases are presented in Table 1, and

some are very different from one gas to another. For example, hydrogen (H_2) has a sound speed 4 time greater than the one of air, its thermal conductivity is 7 times greater, its viscosity 2 times smaller and its mass density 14 times smaller.

Table 1. Physical properties of common industrial gases [2-4]

Gas	Mass density (kg/m^3) 1.015 bar, 15°C	Viscosity ($\mu Pa.s$) 1.015 bar, 0°C	Thermal conductivity ($mW.m^{-1}.K^{-1}$) 1.015 bar, 0°C	Sound speed ($m.s^{-1}$) 1.015 bar, 20°C
CO_2	1.87	13.7	14.7	267
O_2	1.35	19.1	24.4	326
Air	1.22	17.2	24.4	343
N_2	1.18	16.6	24.0	349
CH_4	0.68	10.1	30.6	446
He	0.17	18.7	146.2	1007
H_2	0.09	8.4	172.6	1270

The detection of gas based on the measurement of gas physical properties do not need a sensitive layer. Then such sensors avoid having to choose or develop a new sensitive layer for each application depending on the species to be detected and the possible interferents [5] (*bottleneck b*). Moreover, such sensors become generic sensors and can address different niche markets without any long development process (*bottleneck a*). Finally, the absence of a sensitive coating, which is subjected to sorption or redox phenomena in classical chemical sensors, leads to a more reliable and reversible behavior (*bottleneck c*).

Examples of such physical sensors for gas detection are thermal conductivity sensors [6-8], mass density and viscosity sensors [9-10], speed of sound sensors [11]. Commercialized devices also exist based on mass density [12] or thermal conductivity [13] measurements. Due to their physical principle, such sensors achieve good reliability and almost no drift. Their major drawback is that there is no selectivity nor discrimination because there is only one physical parameter measurement. Consequently, they are limited to determination of the concentration of a known binary mixture.

In this chapter, examples of physical sensors for gas detection using vibrating MEMS are presented. The presented results have been recently (2019-2022) realized in the IMS laboratory in collaboration with other French laboratories (GREMAN, LAAS, CRHEA). Moreover, it is shown how combining multiple physical properties measurement it is possible to discriminate different gas mixtures and to determine the gas concentration. In section 2, the vibrating MEMS are silicon microcantilevers, whereas in section 3 they are silicon nitride capacitive micromachined ultrasonic transducers. Then, section 4 presents how using such sensors (microcantilevers or CMUT) it is possible to replace the selectivity, usually obtained with the use of a sensitive coating, by discrimination of different gas mixtures. Finally, section 5 presents some potential applications of such sensors.

2. Detection using microcantilevers without sensitive coating

The operational principle of an uncoated resonant microcantilever utilized as a chemical sensor relies on the influence exerted by the mass of the fluid displaced by the oscillating cantilever on its resonant frequency. Indeed, when the density of the surrounding fluid increases (decreases), the effective equivalent mass of the cantilever also increases (decreases), thus leading to a reduction (increase) in the resonant frequency. In the context of chemical detection in gaseous environments, variations in gas density can serve as an indicator of alterations in the concentration of specific chemical species within a gas mixture [14-16].

The outcomes presented herein were achieved using silicon microcantilevers featuring electromagnetic actuation and piezoresistive read-out (Figure 1):

- The process of generating vibrations entails passing alternating current (AC) through the conductive wire situated along the cantilever's periphery. When a magnetic field aligned with the longitudinal axis of the beam is present, it generates an AC Lorenz force at the microcantilever's free end, subsequently inducing vibrations out of the plane.
- To effectively capture these vibrations, the process incorporates the creation of two semiconductor strain gauges (boron-doped piezoresistors). They are organized in a semi Wheatstone bridge setup: one gauge is positioned at the point of maximum strain (on the surface of the clamped-end of the microcantilever), while the second gauge is positioned on the rigid substrate.

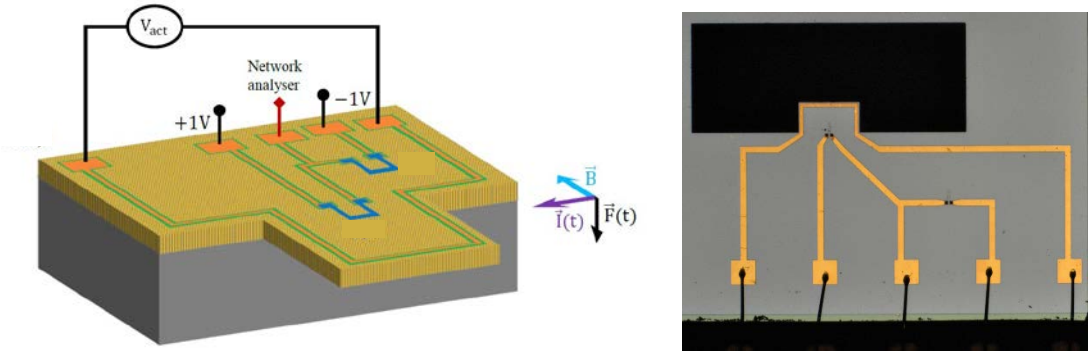

Figure 1. *Schematic representation and photo of the microcantilever with electromagnetic actuation and piezoresistive read-out.*

To assess the impact of cantilever geometries (including rectangular, U-, and T-shaped microstructures) and dimensions, a series of microcantilevers were manufactured at LAAS (Toulouse, France) and subjected to testing at IMS (Bordeaux, France) [17]. The results demonstrated that for mass density measurement, uniform rectangular cantilevers exhibited superior performance compared to their T- and U-shaped counterparts. Additionally, the investigation revealed that broader and shorter beams manifested heightened sensitivity toward changes in mass density. The sensitivity of rectangular beams was found to follow a proportional relationship with b/L^2 (where b and L represent width and length, respectively), in accordance with the analytical model developed by IMS [14]. Interestingly, the thickness of the rectangular cantilevers didn't influence their sensitivity to mass density changes, despite its effect on the resonant frequency. Nevertheless, the noise on the resonant frequency is dependent on the microcantilever's thickness, underscoring the need for careful consideration when selecting the most suitable thickness for a given structural size (length and width). This consideration becomes especially relevant for optimizing the limit of detection.

Owing to the minor shifts in resonant frequency that require detection, novel characterization techniques have been developed to monitor these subtle frequency variations. The traditional approach to track changes in resonant frequency involves the identification of the resonant peak within the amplitude spectrum and tracing alterations in the associated frequency. However, the relative frequency variation for scenarios involving low hydrogen (H_2) concentration in nitrogen (N_2) is exceedingly small. For example, when utilizing a silicon microcantilever featuring dimensions of 1 mm length, 1 mm width, 10 µm thickness, and an approximate resonant frequency of 23kHz, a concentration as low as 0.2% of H_2 in N_2 yields a resonant frequency shift of about 0.25 Hz (equivalent to a 10 ppm resonant frequency variation). As a result, the conventional technique proves inadequate in capturing such minute frequency shifts accurately. Primarily, this is attributed to the challenge of accurately pinpointing the exact location of the resonant peak due to inherent measurement noise. Consequently, alternative characterization methods involving the analysis of gain and phase spectra have been experimented using measurement data [18]. Derived from the same dataset, the diverse methodologies used to extract resonant frequency yielded different signal-to-noise ratios, spanning from 1.2 for the conventional technique to 190 for the most effective strategy (which involved

linearizing the phase around the resonant frequency). The results presented in this section predominantly rely on the application of phase linearization, often employing a silicon cantilever measuring 1 mm x 1 mm x 10 µm (with a resonant frequency of around 23 kHz and a quality factor of approximately 500 in atmospheric pressure).

To conduct gas detection, the microcantilever is positioned within a tightly sealed gas chamber, with a volume of 500 µl, and subjected to controlled gas flow conditions. Gas compositions containing binary mixtures featuring the desired elements (such as hydrogen, helium, carbon dioxide, or methane) and nitrogen are introduced into the chamber, employing gas bottles and a suite of mass-flow controllers (Brooks 5850S). Each binary mixture is systematically introduced at three distinct concentrations—namely, 5%, 4%, and 3%—for a time span of 400 seconds each. Between the introduction of each gas mixture, nitrogen is introduced for a 400-second interval, serving to assess potential drift and to underscore the sensor's reversibility. The resonant frequency shifts observed throughout this experiment are depicted in Figure 2.

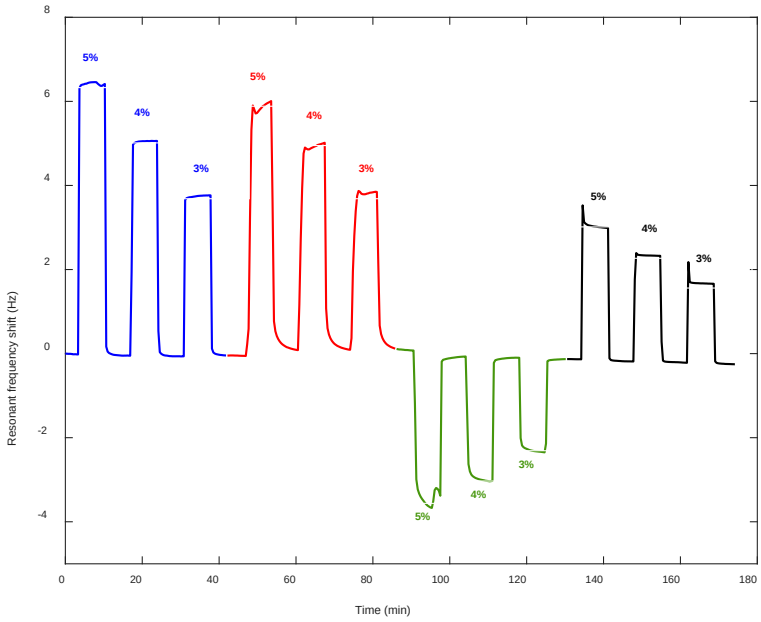

Figure 2. *Example of resonant frequency shifts measured with a 1 mm x 1 mm x 10 µm silicon cantilever with different concentrations (5%, 4%, 3%) of H_2 in N_2 (blue curve), He in N_2 (red curve), CO_2 in N_2 (green curve) and CH_4 in N_2 (black curve) with a gas flow of 100ml/min at room temperature conditions (temperature ≈ 20°C, pressure ≈ 1 atm).*

Based on the physical principles governing these sensors, the presence of gases lighter than nitrogen (such as H_2, He, and CH_4) results in an increase in the resonant frequency, while the presence of heavier gases (like CO_2) than nitrogen leads to a decrease in the resonant frequency. Furthermore, the trends match the full reversibility and quick response time (under 1 minute). In our experiment, the primary factor influencing response time is the duration required for the flow feedback loop to stabilize concentration levels (a swifter control system could potentially achieve sub-1-minute response times). Additionally, almost no drift is observed over the experiment's duration (a mere 0.15 Hz over 3 hours without room temperature control). Of notable significance is the consistent shift in resonant frequency, surpassing 2 Hz for the tested concentrations (5%, 4%, and 3%) across all four gases (equivalent to 100 ppm of the resonant frequency). Moreover, the capability to measure even lower concentrations is facilitated by the minimal noise associated with measurements based on phase linearization (an exemplar of detecting 100 ppm of H_2 in N_2 is depicted in Figure 3).

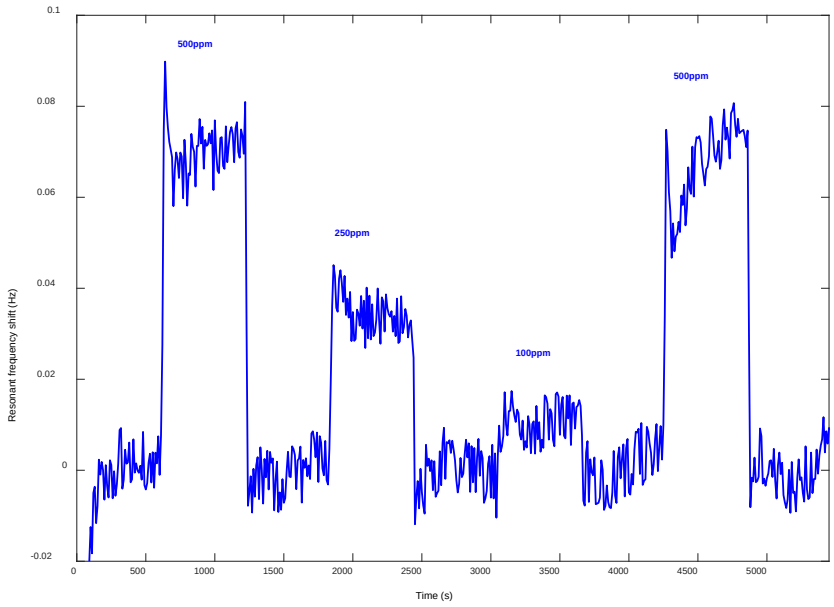

Figure 3. *Example of resonant frequency shifts measured with a 1 mm x1 mm x 5 µm silicon cantilever with small concentrations (500ppm, 250ppm and 100ppm) of H_2 in N_2 with a gas flow of 100ml/min at room temperature conditions (temperature ≈ 20°C, pressure ≈ 1 atm).*

As proved with the measurements presented in Figure 2, the resonant frequency measurement allows to discriminate if the gas becomes heavier or lighter but there is no selectivity between the different gas mixtures (He, H_2 and CH_4 can be confused with each other). Concerning this point, the measurement of another parameter (the quality factor) allows to discriminate between the different gas mixture (section 4).

3. Detection using CMUT without sensitive coating

Another type of MEMS has been used to achieved chemical detection using gas physical properties measurement: silicon-based capacitive micromachined ultrasonic transducers (CMUT), which consist of small membranes that can be electrostatically actuated and the vibration can be measured with capacitive read-out.

The presented results have been achieved using silicon nitride CMUT microcantilevers fabricated at GREMAN (Tours, France) using the surface machining process described in [19]. Each CMUT sensor (8 mm x 0.8 mm) is composed of thousands of single CMUT in parallel (Figure 4). Each membrane is a square (32 µm x 32 µm) of 450 nm thickness. The resonant frequency is around 8 MHz.

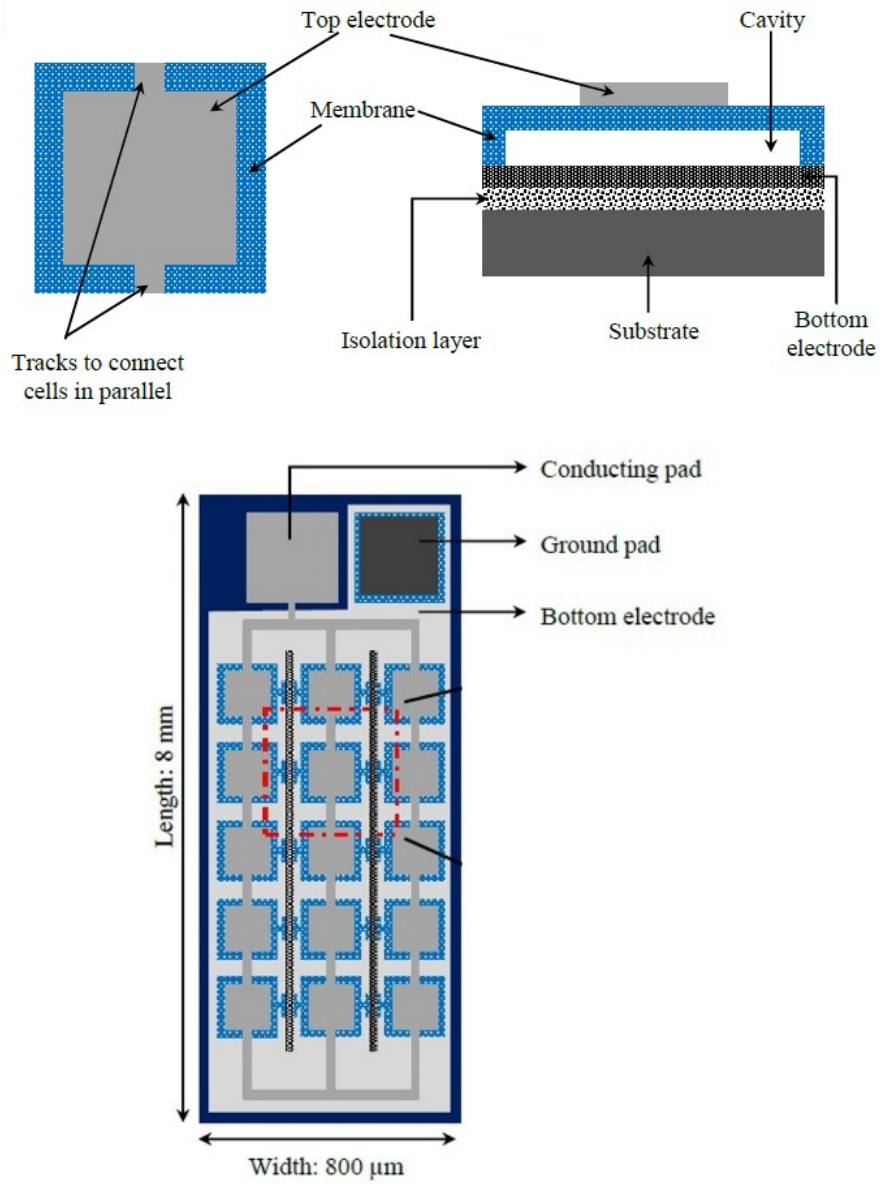

Figure 4. *Schematic representation of the CMUT membrane and CMUT chip.*

The same principle of gas detection as the one presented using microcantilever (section 2) based on the spectrum measurement around resonant frequency has been used. In this case, the measurement is based on the measurement of the spectrum of the CMUT admittance. It has been proved that the more sensitive measurement is obtained with the measurement of the minimum of the admittance [20]. Examples of detection of H_2 or CO_2 in N_2 are presented in Figure 5. Similar conclusions can be drawn from those in section 2.

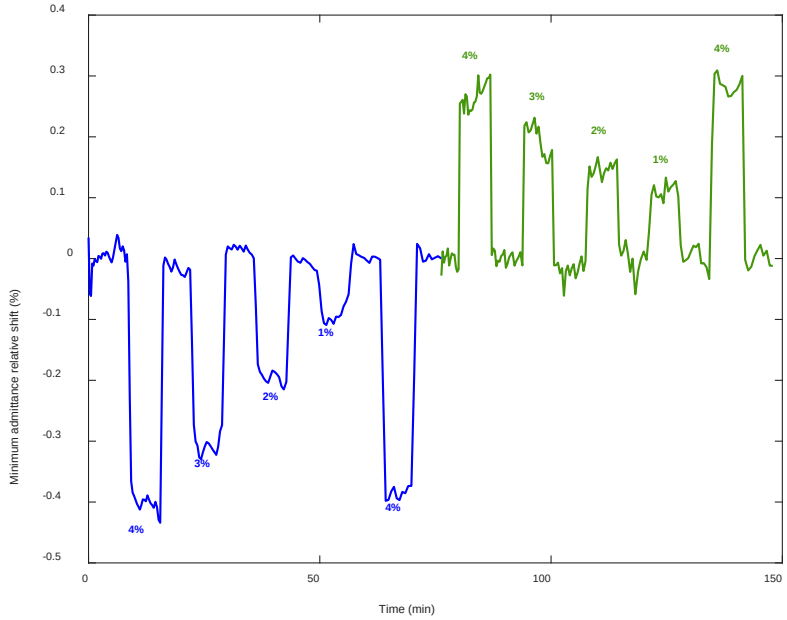

Figure 5. *Examples of gas detection using the measurement of the relative variation of the admittance of a silicon nitride CMUT composed of squared membranes (32 µm x 32 µm) on a 8 mm x 0.8 mm chip. The measurements have been made at different concentrations (4%, 3%, 2%, 1%) of H_2 in N_2 (blue curve) or $C0_2$ in N_2 (green curve) with a gas flow of 100ml/min at room temperature conditions (temperature ≈ 20°C, pressure ≈ 1 atm).*

Besides, two other innovative uses of CMUT for the gas detection have been also demonstrated: the first one consists in measuring the propagation time between two CMUTs and the second one consists in measuring the attenuation of the acoustic wave between two CMUT [21-23].

The basic way to measure the time of flight between two CMUT consists in measuring the time that an ultrasound takes to propagate from a CMUT in emission mode to a CMUT in reception mode, both placed in front of each other. An ultrasonic pulse is sent through the gas mixture by a CMUT in emission mode. After travelling a distance of few millimeters, it is then received at the other end by another CMUT in reception mode (Figure 6). Both the emission and the reception signals were acquired via a Picoscope2000 Series acquisition card and then analyzed through a basic peak detection algorithm.

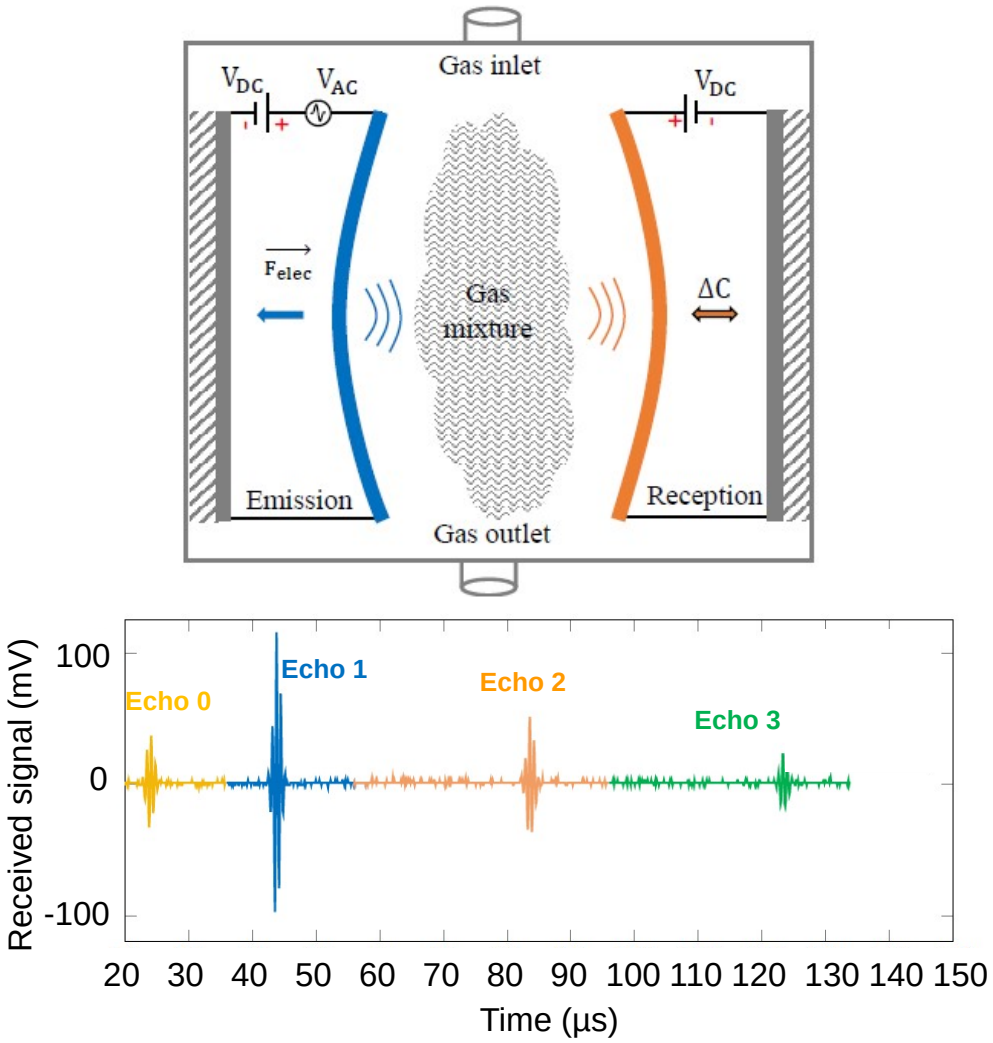

Figure 6. *Schematics illustrating the principle of a time of flight measurement for gas detection with 2 CMUT and example of measurements of echoes in nitrogen gas*

Examples of detection of H_2 (sound velocity higher than the one of N_2) or CO_2 (sound velocity lower than the one of N_2) in N_2 are presented in Figure 7. The measurements are in good agreement with analytical modeling [21]. As for the other measurements presented in this chapter, they are reversible, with almost no drift and response time below 1 min.

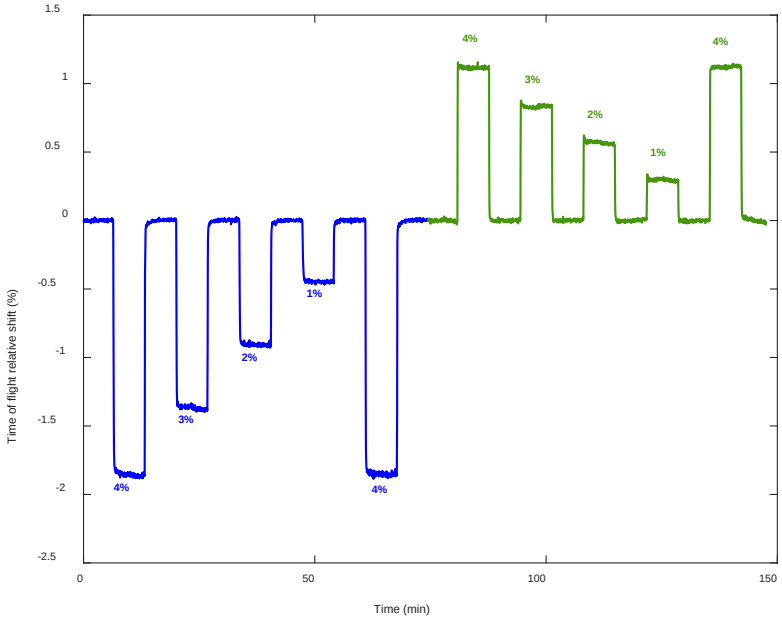

Figure 7. *Examples of gas detection using the time domain measurement of the relative shift of the time of flight between two CMUT. The CMUT are silicon nitride CMUT composed of squared membranes (32 µm x 32 µm) on a 8 mm x 0.8 mm chip. The measurements have been made at different concentrations (4%, 3%, 2%, 1%) of H_2 in N_2 (blue curve) or $C0_2$ in N_2 (green curve) with a gas flow of 100ml/min at room temperature conditions (temperature ≈ 20°C, pressure ≈ 1 atm).*

Although intuitive and effective, the measurement of the time of flight in the time domain presents some drawbacks. One of them being the need for a robust peak detection algorithm which usually is hard to integrate in a sensor and also required computational cost. Another issue is that a pulse generator is usually more expensive than a simple sine wave generator. An alternative method to measure the time of flight is to use the same setup (2 CMUT facing each other) but, instead of generating pulse, sinusoidal wave is emitted and received. In this case, the time of flight is the slope of the phase between emitted and received signals as a function of the radial frequency. A data processing was set up in order to suppress the influence of the multiple echoes [24].

Examples of the same detection as those of Figure 7 are presented in Figure 8 with the time of flight measurement based on the spectrum of the phase. It can we easily seen that this method of measurement is less noisy.

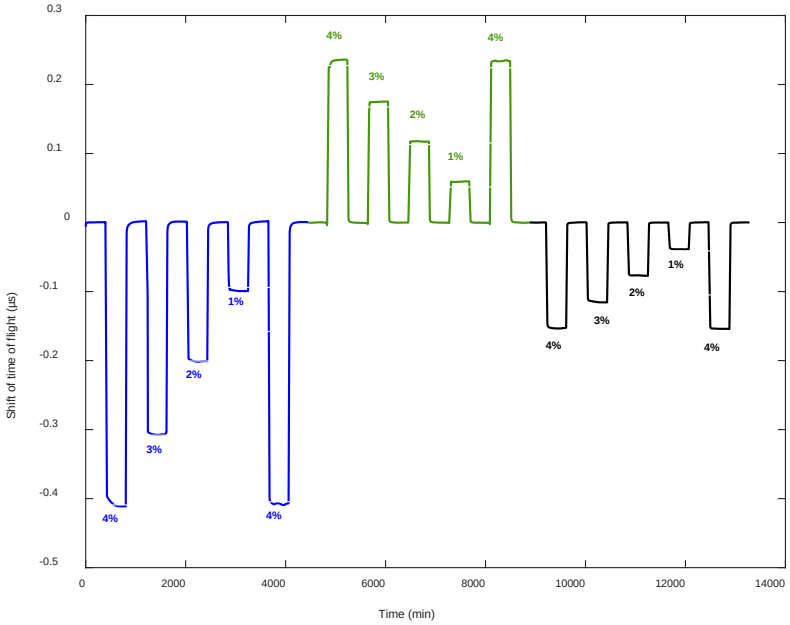

Figure 8. *Examples of gas detection using the frequency domain measurement of the shift of the time of flight between two CMUT. The CMUT are silicon nitride CMUT composed of squared membranes (32 µm x 32 µm) on a 8 mm x 1 mm chip. The measurements have been made at different concentrations (4%, 3%, 2%, 1%) of H_2 in N_2 (blue curve) or $C0_2$ in N_2 (green curve) or CH_4 in N_2 (black curve) with a gas flow of 100ml/min at room temperature conditions (temperature ≈ 20°C, pressure ≈ 1 atm).*

Another physical property of the gas that can be measured with the same setup is the sound attenuation of the gas. Indeed, instead of measuring the delay that the acoustic wave takes to travel the cell, the amplitude of the sinusoidal signal amplitude can be measured: an increase in the gas attenuation decreases the amplitude of the signal. The advantage of the attenuation is that it is a frequency dependent property and its dependence varies from one gas to another. For example, the attenuation at 4 MHz is much higher for H_2 than for CO_2 or CH_4. As shown in Figure 9, an appropriate choice of the frequency measurement can achieve a kind of selectivity: MHz range is appropriate to detect hydrogen with small interference with other gases.

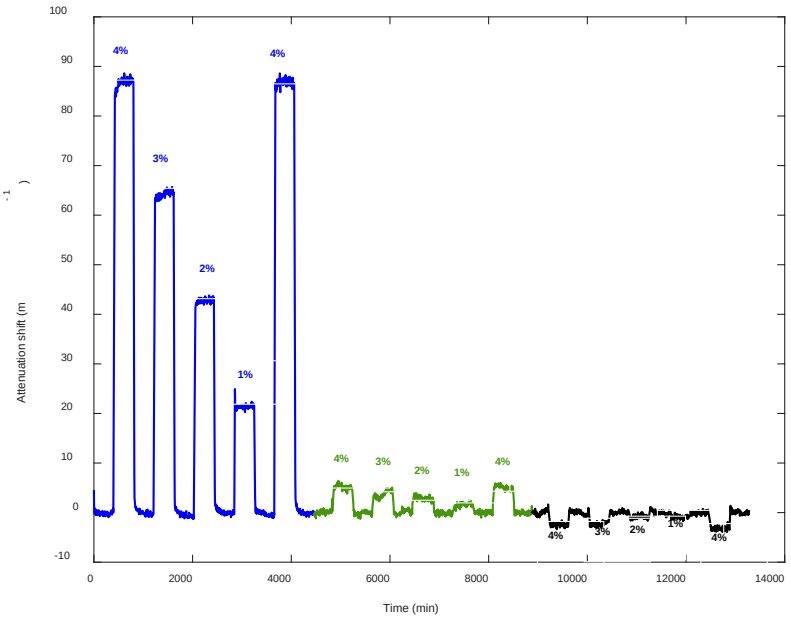

Figure 9. *Examples of gas detection using the frequency domain measurement of the attenuation shift between two CMUT at 4MHz. The CMUT are silicon nitride CMUT composed of squared membranes (32 µm x 32 µm) on a 8 mm x 0.8 mm chip. The measurements have been made at different concentrations (4%, 3%, 2%, 1%) of H_2 in N_2 (blue curve) or $C0_2$ in N_2 (green curve) or CH_4 in N_2 (black curve) with a gas flow of 100ml/min at room temperature conditions (temperature ≈ 20°C, pressure ≈ 1 atm).*

As the microcantilever-based measurements presented in section 2, the CMUT-based measurements of this section are not selective: the time of flight measurement allows to discriminate if sound velocity of the gas increases or decreases and the attenuation measurement allows to achieve a partial selectivity by appropriate choice of frequency. Combining time of flight measurement with attenuation measurement allows to discriminate between the different gas mixture (section 4).

4. Discrimination of gases with multiple physical property measurements

As presented in section 2 and 3, resonant MEMS can be used to measure multiple physical properties of gas. One physical property measurement is not sufficient to achieve discrimination between different gases. Using the resonant MEMS spectrum, it is possible to measure simultaneously two gas physical properties and then to achieve discrimination between different gas mixtures and to measure their concentration. This will be shown in this section with both the microcantilevers and the CMUT of previous sections.

Using the spectrum measurement not far from the microcantilever resonant frequency, it is possible to measure both the resonant frequency and the quality factor of the microcantilever. Using a simple model [25] it is then possible to extract the relative variation of both the mass density and viscosity of the gas. In the plane (mass density, viscosity), the measurement points are not far from the one expected by theory (Figure 10). Then, depending where the point is in this plane the discrimination between different gas mixtures can easily be made and after that the mass density measurement, which is less noisy than the viscosity measurement, can be used to estimate the gas concentration.

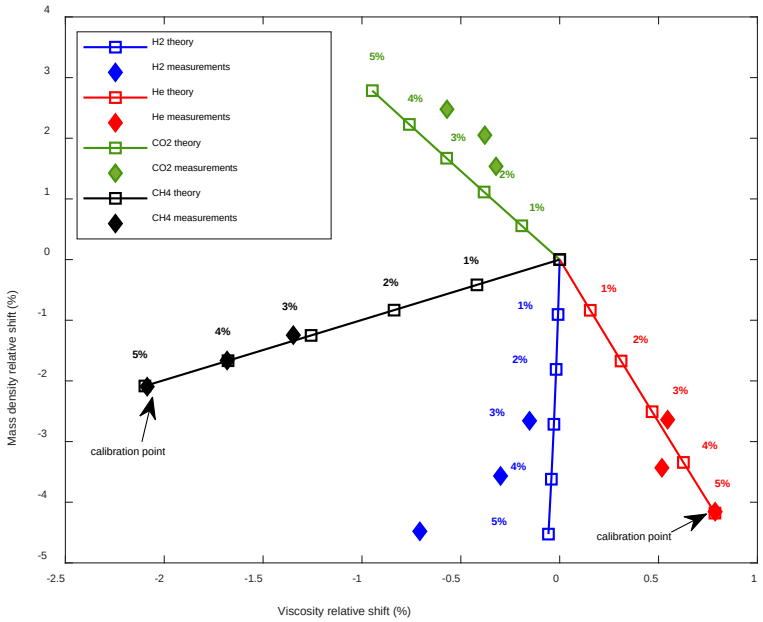

Figure 10. *Simultaneous measurements of both the mass density and the viscosity of the gas using the simultaneous measurement of both the resonant frequency and quality factor of a silicon cantilever (1 mm x 1 mm x 10 µm) with different concentrations (5%, 4%, 3%) of H_2 in N_2 (blue), He in N_2 (red), $C0_2$ in N_2 (green) and CH_4 in N_2 (black) with a gas flow of 100ml/min at room temperature conditions (temperature ≈ 20°C, pressure ≈ 1 atm). The theoretical points are also reported.*

On the same principle using the spectrum measurement of the signal of the receiver CMUT, it is possible to measure both the time of flight and the attenuation at each frequency (Figures 8 and 9). As in the latter case (Figure 10), depending where the point is in the plane (attenuation, time of flight) the discrimination between different gas mixtures can easily be made and the gas concentration can be deduced (Figure 11).

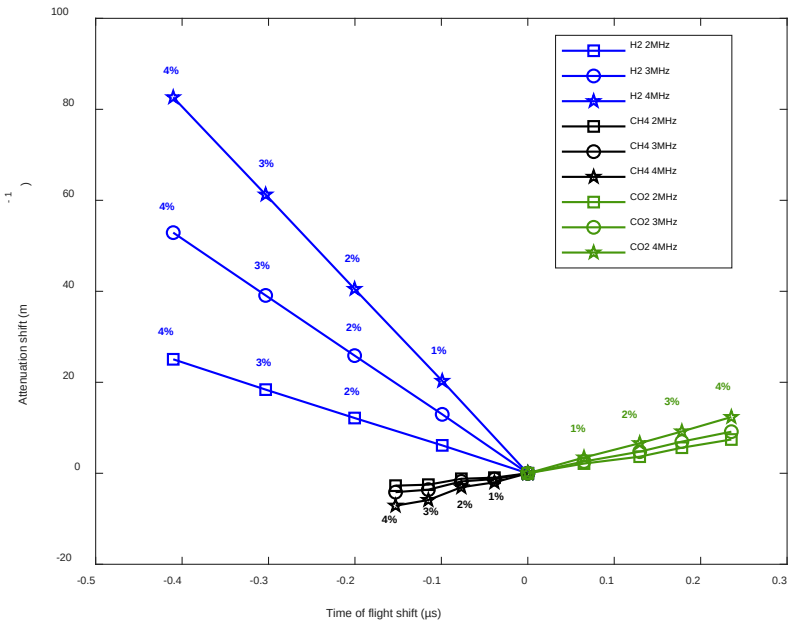

Figure 11. *Simultaneous measurements of both the time of flight and the sound attenuation of the gas using the simultaneous measurement of both amplitude and phase of the signal of the CMUT receiver with different concentrations (5%, 4%, 3%) of H_2 in N_2 (blue), He in N_2 (red), $C0_2$ in N_2 (green) and CH_4 in N_2 (black) with a gas flow of 100ml/min at room temperature conditions (temperature ≈ 20°C, pressure ≈ 1 atm).*

5. Potential applications

The presented sensors do not target a particular application, but, as the developed devices are generic, it is possible to use this kind of devices for different applications where quantitative determination of non-reactive gas compositions in gas mixtures requires particular attention. Such sensors could be of useful interest for a wide range of industries for the continuous monitoring of gases. Important markets include gas production, hydrocarbon fuel distribution, biogas production and distribution. Few possible applications are listed in more detail below:

- With the global population consistently on the rise, there is an exponential surge in the demand for diverse goods and services. This unfolding scenario of industrial expansion, coupled with the rapid growth of the populace, amplifies the need for energy, necessitating its provision through both traditional and renewable energy sources. Sustainable energy solutions have undoubtedly become the cornerstone of many contemporary scientific and technological advancements. Scientists are tirelessly seeking solutions that won't contribute to the prevailing carbon emissions. Within this context, it's noteworthy to highlight the widespread adoption of hydrogen-based energy generation as a means of securing clean energy. Hydrogen gas possesses an exceptional energy density. However, given the highly explosive nature of hydrogen-air mixtures within the 4%-75% hydrogen concentration range, continuous monitoring of hydrogen concentration becomes paramount. This is especially crucial due to the potential risks associated with the production, storage, and transport of hydrogen gas. Consequently, vigilant monitoring of hydrogen gas concentration remains indispensable to avert potentially hazardous situations.
- The forthcoming French repository for radioactive waste is planned to encompass underground structures designed to house tens of thousands of cubic meters of high-level and intermediate-level long-lived radioactive waste, predominantly originating from French nuclear power plants. The CIGEO project aims to confine these waste products for an extended period, with the overarching objective of ensuring sustainable, secure, and reversible storage [26]. Central to the comprehensive monitoring strategy for geological disposal is the continuous observation and assessment of subsurface installations. The monitoring apparatus within such an environment must withstand the harsh conditions prevalent in a repository, particularly the radiation levels that could accelerate the deterioration of sensor materials. The adaptation of existing sensors or the creation of new ones becomes imperative to align with the technical demands of this application. This significance is especially pronounced in the domain of chemical sensors, where enhancing durability and minimizing maintenance requirements are paramount. In this context, the release of hydrogen is anticipated within the radioactive waste disposal facility, arising from both (i) the release of radioactive waste and (ii) the anoxic corrosion of metallic components. Consequently, as in the previous scenario, it becomes imperative to actively monitor the concentration of hydrogen gas within this radioactive repository, thereby averting any potential hazardous circumstances.
- Serving as a premium-grade industrial raw material, hydrogen finds extensive applications not only in industries such as nuclear, aerospace, and metallurgy, but also beyond. The molecular structure of hydrogen is incredibly compact, endowing it with exceptional permeability. Consequently, the minute gas molecules of H_2 are inclined to escape through even the most minuscule openings and fissures. As a result, the detection of hydrogen leakage emerges as a critical concern within a multitude of industrial domains.

- The same problem of leakage in small holes or permeable materials could happen with helium gas which is also composed of very small molecules. As described in [27] it is important to avoid exposing smartphones or smartwatches to helium, because the small gaseous molecules can bleed into the MEMS devices and stop them from functioning. It is then important to control the concentration level of helium if helium is present not far.
- Since the liberation of the French gas market, the importance of fuel gases (i.e. natural gas) in industrial applications has increased significantly. Non-traditional sources result in a gas composition that can vary greatly. Sources of gas can be traditional dry gas from natural gas wells, wet shale gas, biogas and synthetic natural gas made from a blend of propane and air. Significant variation of gas composition and energy content (combustion potential) creates added complexities in the design of burner control systems. The percentage of non-combustible gas (CO_2 and N_2) in fuel gas can vary from 1% to 20% depending on the source. There is then a need to measure the gas composition of fuel gas.

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