The Capactive Micromachined Ultrasonic Transducer (CMUT) as a Chem/Bio Sensor

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Abstract— Airborne Chem/Bio sensors increase in importance every day for applications in homeland security for the detection of bio-hazardous materials and improvised explosive devices (IED); in the spoilage of food; in the health care industry for detecting cancer, diabetes, and other conditions; and in many other industries. A number of ultrasonic sensors have been developed and used in the Chem/bio sensing arena: quartz crystal micro balance (QCM), surface acoustic wave (SAW) resonators, and more recently resonant and simply deflected cantilevers.

The capacitive micromachined ultrasonic transducer (CMUT) is a "platform" device that has been investigated in many airborne and immersion ultrasound applications. In this work, we explore the use of the CMUT as a Chem/bio sensor. Because the membrane of a CMUT can be a fraction of a micron thick, and the frequency of operation in the MHz range, it is possible to obtain sensitivity in the order of 1 femto-gram mass loading per cell with proper design. Another important attribute of the CMUT as a Chem/bio sensor is that many cells are used to make a sensor which helps improve its false alarm rate over existing sensors where only one resonant element is used. Finally, a high mechanical quality factors (Q) realized in CMUTs which results in very low noise floor, which is necessary to make a very sensitive sensor.

An array of CMUTs was made resonant in air at 6 MHz with a Q of 160 and a noise floor of 0.4 Hz in a 1 Hz bandwidth, and where each element consisted of 750 cells. Each cell had a diameter of 30 microns and a silicon nitride thickness of 0.85 microns. Six elements of the array were functionalized with different polymers (PAAM, PEG, PSS, PVA) and the sensitivity of the sensors was measured by flowing different analytes (water, ethanol, isopropyl alcohol, toluene) at different concentrations over the sensors. The frequency shift of each resonator was measured using a frequency counter. The sensor had a volume detection sensitivity of 20 ppb, and a calculated mass loading sensitivity of 1 femto-gram. We will also present results from a new generation of sensors operating in the 25 MHz frequency range and with an expected sensitivity improvement of one to two orders of magnitude over the earlier design.

Keywords-component; capacitive micromachined ultrasonic transdcuer; sensor; resonator; polymer

Introduction

Chemical/biological sensors are necessary and important in many applications. Most urgent today is the ability to improve the detection of improvised explosive devices and chemical warfare agents. Other applications include monitoring of food and beverage products, health care, and perfume and wine industries to name a few. A number of ultrasonic physical devices have been combined with thin layers of polymers and used as chemical/biological sensors for these applications; namely the quartz crystal oscillators (QCM), surface acoustic wave resonators (SAW), Lamb wave resonators, cantilevers, and more recently film bulk acoustic wave resonators (FBAR). All these sensors detect the presence of an agent as it is absorbed by the polymer and mass loads the sensor, resulting in a frequency shift. In this work we discuss the use of the capacitive micromachined ultrasonic transducers (CMUT) as a mass sensor that is functionalized with polymers in the same fashion as other ultrasonic sensors. We will report on the realization and characterization of the CMUT as a sensor and show that it has potential advantages over existing sensors in terms of sensitivity, low false alarm rate, robustness, ease of electronic integration, and cost [1].

CMUT as a chemical/biological sensor

A. What is a CMUT

The capacitive micromachined ultrasonic transducer (CMUT) consists of a combination of capacitor cells that are all connected in parallel. A single cell is shown in cross section in Fig. 1 where the membrane can be made of silicon or silicon nitride or any of a variety of materials.

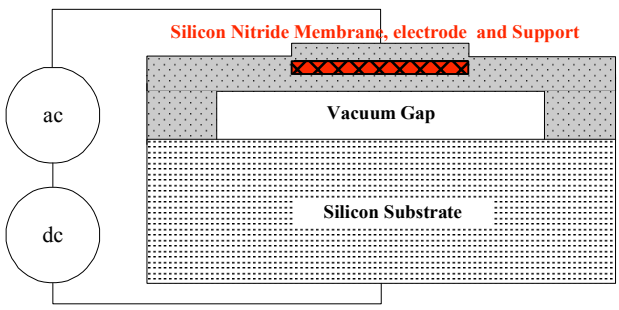

Figure 1 Schematic diagram of a CMUT cell.

In typical operation, a DC voltage is applied between the top and bottom electrodes to provide an electric field in the gap. This electric field provides the electromechanical coupling

of the capacitor transducer, and the strength of this electric field is directly proportional to the strength of the electromechanical coupling of the capacitor transducer.

The membrane acts as a resonant member whose resonant frequency depends on the thickness of the membrane or it's mass. Hence, if the membrane is loaded by a mass, the resonant frequency of the membrane will change according to the size of this mass. It is this effect that is used in this work, along with the chemical functionalization, to turn this physical sensor into a chem/bio sensor. To get a measure for the potential sensitivity of a resonant membrane, we calculate the resonant frequency shift as a function of mass loading of a material with a density of 100 kg/cm^3. For the design of table I, the expected resonant frequency shift as a function of mass loading is shown in Fig. 2.

	50 MHz design
Radius (μm)	6
Thickness (μm)	0.5
Gap (μm)	0.1
Electrode (μm)	5
Collapse (V)	88
Mem. mech. reso. (MHz)	53.05
r_{LOSS}	10 x air loss
Q	130
Bias	80%
Material	Silicon
Insulator	Oxide (0.1μm)

Table I Parameters of a test CMUT sensor cell

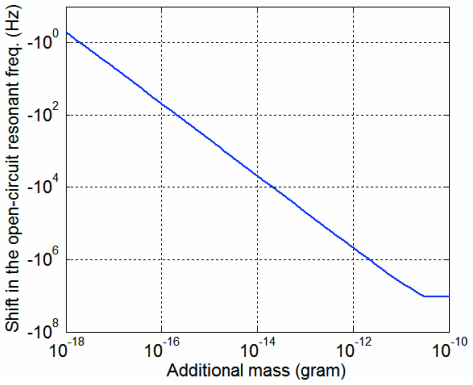

Figure 2 Resonant frequency shift as a function of mass loading for the CMUT cell of table I.

It is clear from Fig. 2 that when a CMUT cell is used as a mass sensor it possesses a large linear dynamic range, and that it is possible to detect a mass loading of 1 atto-gram, at atmospheric pressure, if the noise of the sensor circuit (typically an oscillator) is less than 1 Hz. The results of Fig. 2 are not far from the simple, back of the envelope, estimation of the sensitivity of most mass resonant sensors where we can write:

$$\frac{\Delta f}{f} \approx -\frac{1}{2}\frac{\Delta m}{m}$$

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This indicates that for high mass detection sensitivity, it is necessary to have low noise, high frequency of operation, and resonant structures with low mass.

A typical CMUT is made of a number of cells that are connected in parallel. In this work, the number of cells that are connected in parallel is chosen for best match into a resonant oscillator circuit for lowest noise performance. Another criterion is to keep the size of the sensor as small as possible for minimum mass sensing capability. Besides its sensitivity, a every important aspect of this sensor is that a large number of cells in parallel helps reduce the false alarm rate of the sensor because if one cell fails to register, all the others still will deliver a proper indication of exposure.

B. *Initial results*

An existing medical imaging array was chosen to test the viability of the CMUT as a chem./bio sensor. The existing array had 128 elements and operated around 6 MHz. The cells were made of silicon nitride membranes with cavity height of 0.1 m, and required a DC bias around 10 V for proper operation. Early tests indicated that there was enough mechanical cross-coupling between the elements of the array, and that when integrated with electronics to form a resonant circuit, the resonators were all locked at the same frequency. Dicing the silicon wafer between the elements removed the mechanical cross talk, and it was possible to have several independent sensors that could be functionalized with different polymers. An image of the array with the dicing cuts is shown in Fig. 3.

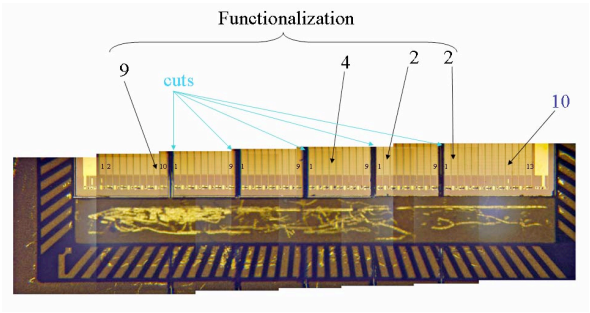

Figure 3 Image of linear array with saw cuts to separate six segments thus allowing the realization of six independent resonant sensors.

Six resonators were constructed with six elements of the array. The resonators had a noise floor of about 0.4 Hz, and a resonant frequency around 6 MHz which could be tuned with the DC bias. The ability to tune the resonant frequency is important in guaranteeing operation at the lowest noise floor for the sensor. Four of the sensors were functionalized with the following polymers: polyaromatic alkylmethacrylate (PAAM), polyethylene glycol (PEG), polysodium 4-styrenesolfonate (PSS), and polyvinyl alcohol (PVA) and were tested with the four analytes: water, ethanol, isopropanol, and Toluene. The testing was done by passing varying concentrations of the analyte in dry nitrogen over the sensors and monitoring the frequency shift. The volume sensitivity was then calculated based on the noise floor of the oscillator, and the mass sensitivity was estimated from theoretical calculation and from

the noise floor of the sensor. Figure 4 shows a sample result of the frequency shift of the sensor with PAAM polymer when water vapor was passed over the sensors in varying concentrations, and in pulses of 30 sec duration. The calculated volume sensitivity of the sensor is found to be 44.6 ppb/Hz which gives an absolute sensitivity of 18 ppb. The mass loading sensitivity is found from the theoretical calculation shown in Fig. 5. For the sensor at a noise floor of 0.4 Hz, the mass loading sensitivity is 1 x 10^{-15} gram/cell, or 0.75 x 10^{-12} gram for the whole sensor as it contained 750 cells. These sensitivities confirm the viability of the CMUT as a chem/bio sensor; especially as new designs that target this application are constructed as opposed to the above tested devices that were designed for medical imaging applications.

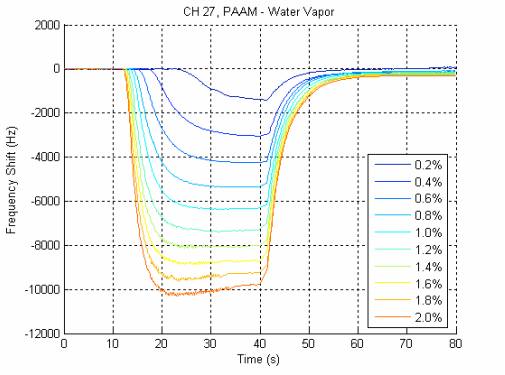

Figure 4 Frequency shift of the PAAM functionalized oscillator as a function of time due to 30 seconds pulses of dry nitrogen with varying concentrations of water vapor.

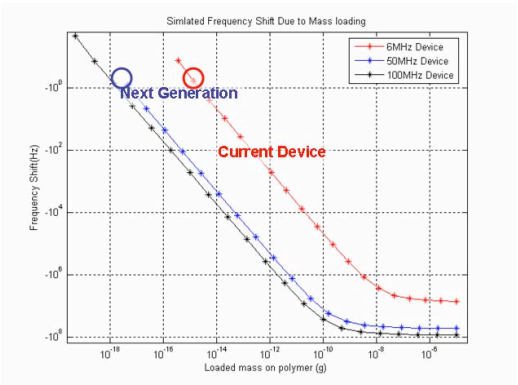

Figure 5 calculated mass loading sensitivity (per cell) of tested device and calculated sensitivity of higher frequency devices with less massive membranes.

C. *New designs*

Fabrication

In order to further improve the sensitivity of the sensor and to enable a large number of independent sensors in a small area, a new generation of devices was designed with a new implementation technology. A manufacturing technology was needed to improve process control, reduce parasitic capacitance, reduce charging effects in metal/oxide structure that are inherent in CMUTs, and reduce the possibility of breakdown which again is always an issue in capacitor devices. The new devices were designed to operate at higher frequency and with less massive membranes in order to improve the sensitivity. No metal was to be used over the membranes of the new generation devices in order to reduce losses due to friction damping and to remove the stressed introduced during metal deposition, and to improve uniformity over an array. Finally, isolation trenches were made between array elements using deep reactive ion etching (DRIE) in order to reduce the cross talk between the elements of the array.

A local oxidation (LOCOS) process was used to define the cavities of the cells in the CMUT transducer. The oxidation process is one of the best understood processes in IC manufacturing, which allows for reliable control over gap height and uniformity over the wafer. The cross section of a CMUT cell that is constructed with the LOCOS process is shown in Fig. 6.

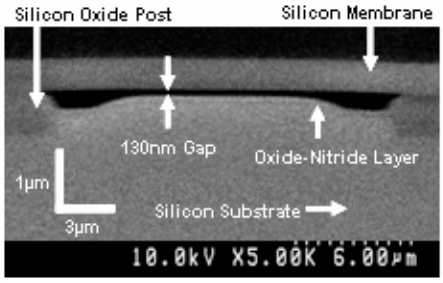

Figure 6 Scanning electron microscope picture of a section through a CMUT cell that is made by the LOCOS process.

A number of important features of this new process are visible in Fig. 6: the oxide post is quite think (~1 m) compared to the oxide-nitride layer and gap height and no metal is deposited on the conductive silicon membrane. The gap of the device of Fig. 6 is 130 nm, while other designs that were realized had gaps of 39.8 nm. It is worth noting that for the design that called for a 40 nm gap, the realized gap was 39.8 nm which demonstrates the control that is afforded by the LOCOS process for making CMUTs.

Characterization

The new devices were characterized by measuring their input impedance as a function of frequency and calculating the quality factor (Q) and maximum phase shift between the open and short circuit resonance. A sample result of the measured input impedance is shown in Fig. 7.

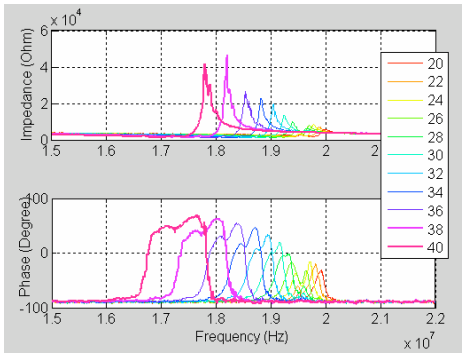

Figure 7 Measured input impedance of new design of a LOCOS produced CMUT.

In the design of Fig. 7, the circular membrane was made of silicon with a thickness of 0.5 m, a gap of 130 nm, a diameter of 18 m, and each device contained 100 cells. The calculated Q of the transducer varied from a low value of 50 at 20 V to a value of 400 at 40 V. The high Q indicates that it should be possible to use these devices in resonator with low noise characteristics. Indeed, the resonators made with these devices operated around 18 MHz with a noise floor of 0.6 Hz which corresponds to an Allan's deviation of:

$$\frac{\Delta f}{f} = \frac{0.6}{18x10^6} = 3.33x10^{-8}$$

This Allan's deviation coupled with the low mass of the resonant membrane indicates that a high mass loading sensitivity will be realized in the sensor.

Functionalization

One element in the array was functionalized with the polymer polyisobutylene (PIB) in order to develop special affinity for the detection of Dimethyl methylphosphonate (DMMP). The PIB was deposited on the transducer using drop on demand technology and a thin coating of about 70 nm was achieved over the whole sensors made of 100 cells.

Sensitivity of new sensor

The new sensor was tested in the same fashion as the previous sensor with 30 second pulses of air with varying concentrations of DMMP. The results are shown in Fig. 8 and the associated volume concentration sensitivity is shown in Fig. 9.

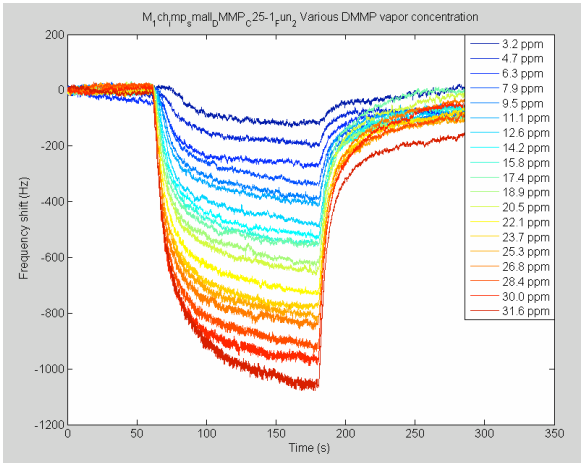

Fig. 8 Frequency shift of the PIB functionalized oscillator as a function of time due to 30 seconds pulses of dry air with varying concentrations of DMMP.

A straight line fit to the data of Fig. 9 indicates that the volume concentration sensitivity is about 38 ppb/Hz which for a noise floor of 0.6 Hz shows a sensor sensitivity of about 23 ppb.

The calculated mass loading sensitivity for this sensor is shown in Fig. 10 and corresponds to a mass loading sensitivity of $2x10^{-17}$ gram/cell or $2x10^{-15}$ gram for the sensor with 100 cells.

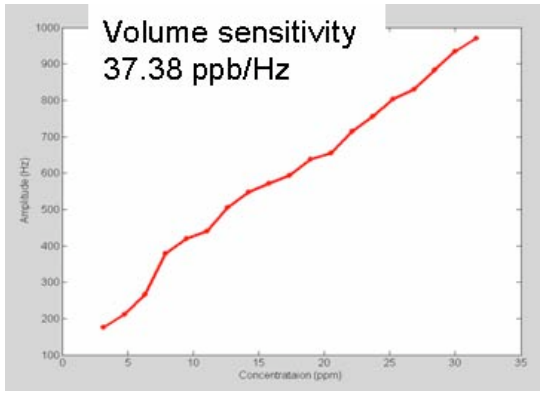

Figure 9 calculated volume concentration sensitivity of the new sensor.

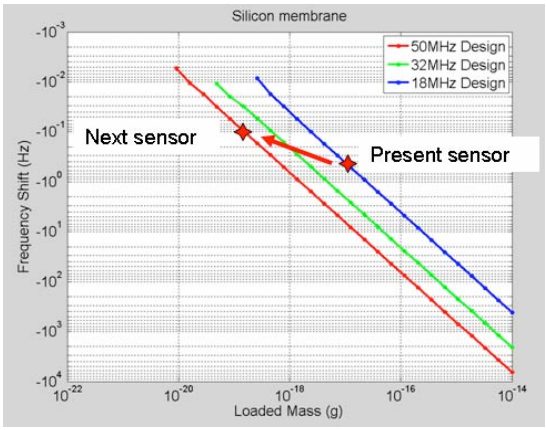

Figure 10 calculated mass loading sensitivity (per cell) of new device and calculated sensitivity of higher frequency devices made in the same processing run.

CONCLUSIONS

It is clear that CMUTs represent a platform technology for many ultrasound applications. The results presented here indicate that the CMUT can be a chem/bio sensor and that the volume sensitivity of this sensor depends very strongly on the chemical functionalization. Further work will include the study of the chemical interaction between polymers and analytes, and that making as many, as orthogonal as possible; sensors will enable the sensitive detection and separation of many analytes.

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