A Low-Power Gas Sensor for Environmental Monitoring Using a Capacitive Micromachined Ultrasonic Transducer

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Abstract— **We present a low-power gas sensor design based on a capacitive micromachined ultrasonic transducer (CMUT), for use on self-powered wearable platforms. Earlier a CMUT-based sensor, with 70-mW power consumption operating at 50 MHz, achieved ppt-level detection limit for chemical warfare agents. In this work we present a sensor operating at 4.33 MHz and consuming 0.77 mW for environmental monitoring. The sensor comprises a polymer-functionalized CMUT resonator in the feedback loop of a Colpitts oscillator. We fabricated the CMUT resonators using a novel process based on anodic bonding. The cavities and bottom electrodes are formed on a borosilicate glass wafer. The device layer of an SOI wafer bonded on glass forms the vibrating plate on top of vacuum-sealed cavities. This fabrication approach reduces process complexity and helps minimize parasitic components. CMUTs with center frequencies in the 3-50 MHz range with Q-factors as high as ~400 have successfully been fabricated. We used a 4.52-MHz device (Q=180) coated with a thin layer of polyisobutylene (PIB) for sensor demonstration.**

Keywords—anoding bonding; capacitive micromachined ultrasonic transducer; environmental monitoring; low-power; mass-loading;

I. INTRODUCTION

In recent years the capacitive micromachined ultrasonic transducer (CMUT) has emerged as a strong candidate for a microelectromechanical resonant-chemical sensor system [1]. There are several advantages offered by the CMUT technology for resonant chemical sensing. The vibrating structure is suspended over a vacuum-sealed cavity and therefore exhibits a higher quality factor compared to cantilevers with similar detection area. Each resonator is composed of hundreds of cells connected in parallel, which ensures robust operation. Multi-channel arrays with elements functionalized with various polymers enhance selectivity. Previously CMUTs were demonstrated as chemical sensors with high sensitivity and high resolution for the detection of chemical warfare agents [2]. In that application power consumption was not the primary constraint. As part of the NSF Nanosystems Engineering Research Center (NERC) for Advanced Self-Powered Systems of Integrated Sensors and Technologies (ASSIST), our goal is to develop low-power, miniaturized, wearable sensors to monitor environmental pollutants continuously and correlate the findings with physiological measurements. For many environmental pollutants high-ppb-to-ppm-level detection is adequate and hence sensitivity can be traded off for power.

We fabricate our devices using anodic bonding process, which has several advantages compared to previously demonstrated fabrication processes for CMUT devices [3]. Wafer bonding in general provides a good control over plate thickness and uniformity by implementing the device layer of a silicon-on-insulator (SOI) wafer as the plate. Anodic bonding is a cost-effective, low temperature bonding process that allows use of patterned metal bottom electrodes on glass substrates to minimize the parasitic series resistance and the parasitic shunt capacitance. Also, the dielectric reliability is improved, as the post region in this approach does not experience any electric field. CMUTs with center frequencies in the 3-50 MHz range with Q-factors as high as ~400 have successfully been fabricated. We used a 4.52-MHz device (Q=180) for the sensor demonstration in the presented work.

The working mechanism of our CMUT resonant sensor is based on mass loading. The CMUT is functionalized with polyisobutylene (PIB), which is sensitive to specific volatile organic compounds (VOCs) such as toluene. When the functionalized device is exposed to a VOC, the additional mass of the absorbed analyte on the resonant structure causes a shift in the resonant frequency. A free-running oscillator designed using the CMUT as the frequency selective device tracks this change in resonant frequency.

The next section describes the CMUT fabrication process and the input impedance characterization results. Section III explains the design and characterization of a Colpitts oscillator with the CMUT resonator in its feedback loop. Initial results on chemical sensing are reported in Section IV.

II. CMUT FABRICATION AND CHARACTERIZATION

A. CMUT Fabrication Process

The CMUT resonators have been fabricated using a novel process based on anodic bonding. The final CMUT structure consists of a vibrating plate, which is composed of a layer of single-crystal silicon and a layer of silicon nitride, and a patterned metal bottom electrode deposited inside a vacuum-sealed, sub-micron cavity. The silicon nitride layer in the structure acts as an insulation preventing electrical shorting

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when the plate comes in contact with the metal bottom electrode.

The starting substrate in our process is a standard 0.7-mm-thick, 100-mm-diameter borosilicate glass wafer (Borofloat® 33, Schott AG, Germany). To implement the vibrating plate of the CMUT we used an SOI wafer that had a 2+/-0.5-mm thick, n-type device layer with 0.001-0.005 Ohm.cm resistivity, a 500-nm-thick buried oxide (BOX) layer, and a 0.5-mm-thick handle wafer with 1-10 Ohm.cm resistivity.

After the borosilicate glass substrate is cleaned with Piranha solution, the cavities are defined with a negative lift-off photoresist. The patterned wafer is hard-baked for 2 hours at an elevated temperature of 125°C in order to get a better adhesion between the photoresist and the substrate before the cavities were etched in 10:1 buffered oxide etch (BOE) solution. 230-nm-deep cavities were etched during 15 minutes total time with 5 BOE-etch cycles of 3 minutes each. An intermediate hard-bake of 10 minutes at 125°C was performed to further strengthen the adhesion of photoresist. The gap height was accurately controlled by depositing 20-nm chromium and 90-nm gold by e-beam evaporation [Fig. 1(a)]. The cavity has a final surface roughness of approximately 2 nm (RMS) after lift-off.

A 200-nm-thick layer of silicon nitride is deposited by plasma-enhanced chemical vapor deposition (PECVD) on the device layer of the SOI prior to bonding. This layer serves as insulation between the top and the bottom electrodes. The anodic bonding is performed at 350°C and 1000-mTorr chamber temperature and pressure, respectively, in a semi-automatic bonding system (Model EVG510, EVG Group, St. Florian, Austria). The voltage is ramped at a rate of 20 V/min and kept at the peak value of 700 V for 30 minutes to prevent insulation layer breakdown. After bonding the handle wafer is ground down to 100 μm and then selectively etched with 10% tetramethylammonium hydroxide (TMAH) at an elevated temperature of 80°C. The BOX layer is removed with 10:1 BOE solution [Fig. 1(b)].

To access the bottom electrode for forming bond pads we etch the plate at the pad location using reactive ion etching (RIE). At this step, the oxygen gas trapped in the cavities during anodic bonding is evacuated. Once the cavities are exposed, oxygen plasma is used for photoresist removal instead of wet cleaning to prevent stiction of the plate on the bottom electrode in the cavities. PECVD silicon nitride with a thickness of three times the gap height is deposited for an appropriate sealing [Fig. 1(c)].

After sealing, the wafer is covered all over with silicon nitride, which is subsequently etched in the bond pad areas and on the active device area. A stack of 20-nm chromium and 130-nm gold is deposited and lifted off in N-Methyl-2-Pyrrolidone (NMP) solvent to implement the bond pads and metallization on the active device area. Fig. 1(d) shows the cross-sectional view of the final fabricated device. The scanning electron microscope (SEM) image of the cross-section of the final CMUT device is shown in Fig.2.

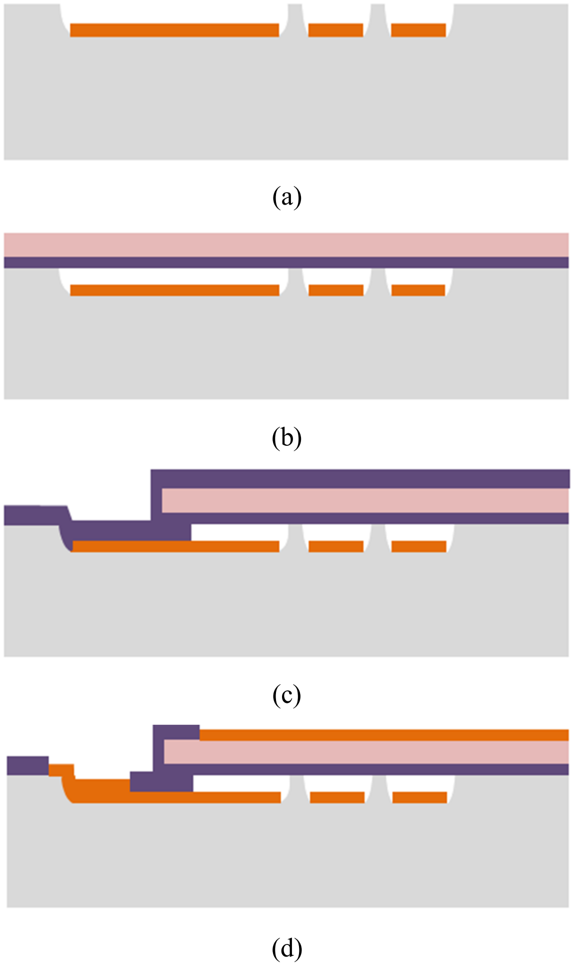
(a)
(b)
(c)
(d)

Fig. 1. Fabrication process flow. (a) After cavity formation and bottom metal electrode deposition; (b) After wafer bonding, and handle and BOX removal; (c) After trapped gas evacuation and sealing by silicon nitride deposition; (d) Final cross-sectional view of a CMUT device (bottom electrodes of a single element are connected. Connections are not shown).

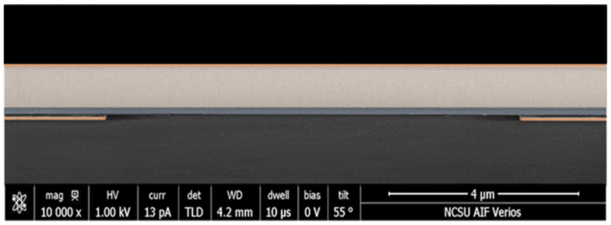

Fig. 2. SEM image of CMUT cross-section.

B. *CMUT Characterization*

A particular CMUT design (Table I) with a parallel resonant frequency of 4.52 MHz is used in this work. The electrical input impedance of this device is characterized in air using a network analyzer (Model E5061B, Agilent Technologies, Inc., Santa Clara, CA) with an internal DC supply available up to 40 V. The input impedance measurement is then fitted to the 6- element Butterworth van-Dyke model with two additional elements R_p and C_p, which takes into account the contact resistance and capacitance, respectively (Fig. 3). The standard Butterworth-van-Dyke circuit consists of four elements, R_m, L_m, C_m, and C_0, which represent the loss, mass, inverse of stiffness, and the electrical capacitance of the resonant structure, respectively [4]. This equivalent model of the CMUT is used in the oscillator design, which tracks the change in the resonant frequency of the sensor in real time.

III. DESIGN AND CHARACTERIZATION OF THE OSCILLATOR CIRCUIT

We designed and built a single-stage tuned Colpitts oscillator circuit, which offers the benefit of low-power consumption, using the 4.52-MHz CMUT as the frequency selective device (Fig. 4). The device under test is shown in Fig. 5.

Table I	
Physical Parameters of the CMUT	
Number of cells per element	240
Length of an element, μm	2390
Width of an element, μm	651.5
Cell diameter, μm	77
Plate thickness, μm	2.1
Gap height, μm	0.12
Insulating layer thickness, μm	0.2
Substrate thickness, μm	700
Bottom metal thickness, μm	0.11
Shape of the cell	Circular

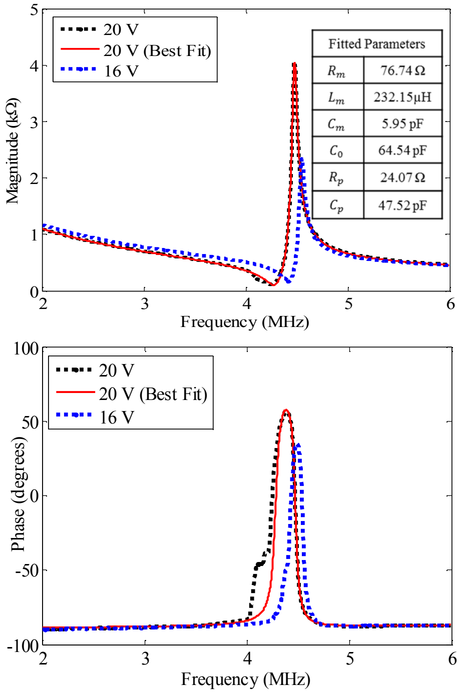

Fig. 3. Measured input impedance of the CMUT (dashed) and fitted curve (solid).

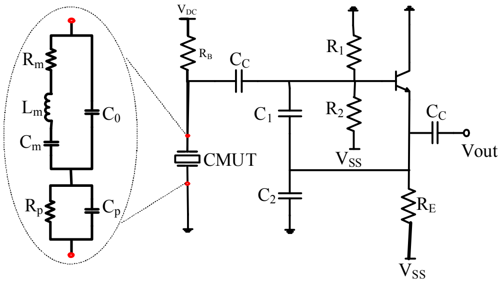

Fig. 4. Schematic of the Colpitts oscillator.

Fig. 5. A group of CMUT resonators (device under test is shown in the box).

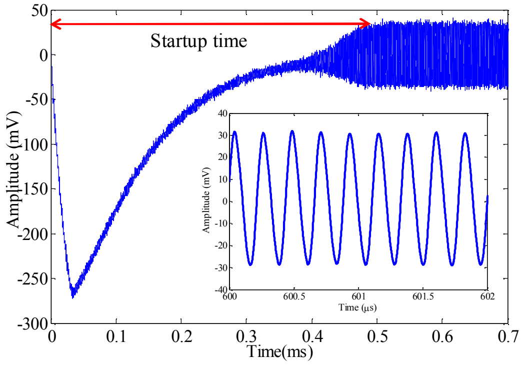

Fig. 6. Output waveform of the Colpitts oscillator.

The basic design idea of the single-stage amplifier is to provide enough gain to compensate for the loss presented by the resonator. The circuit is designed to oscillate at the parallel resonant frequency to take advantage of higher quality factor. The quality factor of the parallel resonance of the CMUT is higher than that of the series resonance as a result of the cell-to-cell non-uniformity [2]. The system does not require an additional start-up circuit as the loop gain is sufficiently larger than unity around the resonant frequency to start the oscillation from in-circuit noise components. The oscillator was implemented on a printed circuit board (PCB) with discrete components. The CMUT is epoxied onto the PCB and directly wire-bonded in order to minimize parasitic capacitance. Oscillation frequency of 4.33 MHz and power consumption of 0.77 mW have been achieved with a -1.5-V supply, when the CMUT DC bias was set at 16 V. The oscillator starts in 0.5 ms after power-up (Fig. 6), which allows low-duty-cycle operation to reduce the average power consumption further.

The short-term frequency stability in the time domain is characterized using a universal frequency counter (Model

53230A, Agilent Technologies, Inc., Santa Clara, CA). The overlapped Allan deviations were computed for different averaging times using the frequency data obtained at a sampling rate of 500 Hz (Fig.7). The curve indicates that a minimum frequency noise of 3.15 Hz (3σ) is achieved at an averaging time of 10 ms.

IV. CHEMICAL SENSING EXPERIMENTS

A. Surface Functionalization

The CMUT device was implemented as a chemical sensor by functionalizing it with polyisobutylene (PIB) for sensing volatile organic compounds (VOCs). Dilute droplets of PIB (1 mg/mL of toluene) were applied on the device using a hand-held adjustable-volume (10 µL – 100 µL range) micro-pipette (Research Plus, Eppendorf AG, Hamburg, Germany). After the solvent dried an approximately 100-nm-thick polymer layer was covering the device. The input impedance of the device was measured after functionalization. We found out that the polymer coating does not significantly perturb the impedance characteristics of the device.

B. Chemical Test Setup

We use a calibration vapor generator (Model OVG-4, Owlstone Inc., Norwalk, CT) to generate National Institute of Standards and Technology (NIST) standard trace concentration level of VOCs. The system generates calibration gas using permeation tube technology at very stable and accurate temperature. The functionalized CMUT was enclosed in a small acrylic glass chamber (3.5 cm^3) and toluene vapor generated by OVG–4 and clean air generated by zero air generator (Model ZAG–6, BCAS Limited, Wallingford Oxon, UK) was flowed into the chamber alternately.

C. Chemical Test Results

We performed gas sensing tests with toluene as a target analyte using the described sensor and experimental setup. Our initial results indicate an average sensitivity 270 Hz/ppm within the range of 10–20 ppm of toluene concentration (Fig. 8). Further experiments with wider range of concentration and different analytes are in progress.

V. CONCLUSION

We demonstrated a low-power resonant chemical sensor based on a 4.52-MHz CMUT resonator fabricated using anodic bonding. Specific achievements reported in this paper include successful implementation of high-quality factor (~180) CMUT resonators, design and implementation of a low-power (0.77 mW) Colpitts oscillator with low frequency noise [3 Hz (3σ)], and initial results on VOC detection by polymer functionalization. Our current efforts focus on further quantification of sensor performance and extension of the demonstrated sensor platform to multiple channels for improved selectivity to detect a variety of VOCs.

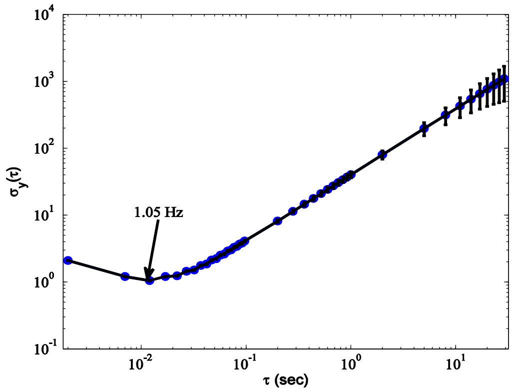

Fig. 7. Plot of overlapped Allan deviation calculated from frequency counter data with a gate time of 2 ms.

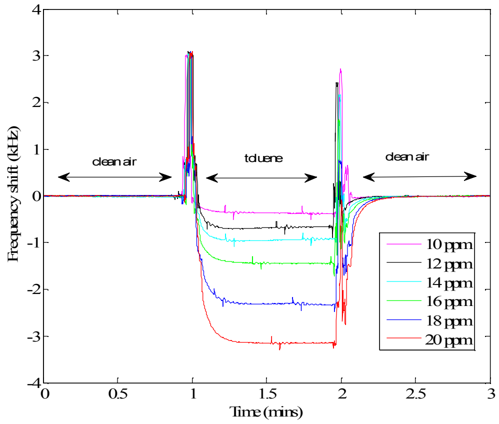

Fig. 8. Chemical test results showing frequency shift in response to toluene (toluene was flowed between 1 to 2 minutes).

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