MEMS Resonant Sensors for Detection of Gasoline Vapor

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Abstract—This work demonstrates detection of organic vapors in the air using micromechanical resonators. Fully silicon thermally actuated resonators with piezoresistive readout have been utilized to detect presence of gasoline vapor in air. The resonators have been fabricated using a single mask process on SOI substrates and have resonant frequencies in the sub-MHz to a few MHz range. Using a thin arbitrary polymer coating as the absorbent layer, frequency shifts in the order of 200ppm were measured for the resonators upon exposure to gasoline vapor. This is equivalent to ~10nm absorbed gasoline thickness. Measured response time constants are around 4 minutes. Faster and more sensitive responses are expected to be achievable using more appropriate absorbent layers and optimized resonator designs.

I. INTRODUCTION

Non-specific sensors capable of detection of organic compounds in gas phase have numerous applications in oil and gas industry among others. Examples of such applications include rapid estimation of oil content of oil sand samples and early detection of hazardous leaks [1,2]. Such sensors can help save plenty of time and resources during oil exploration by avoiding the costly and time consuming process of using off-site laboratory analysis. Previous studies by Clifford [1,2] and Frye [3] have investigated the use of surface acoustic wave (SAW) sensors for detecting volatile organic compounds while Sugimoto [4] investigates the use of conventional quartz crystal microbalance (QCM) sensors for this purpose. Although these studies have investigated the use of real-time micro-sensor based monitoring systems, however, longevity and the impact of variations in environmental variables (e.g., temperature, pressure, relative humidity) on the response are issues which still need to be assessed and resolved for these kinds of sensors.

The intention of this work is to consider MEMS resonant sensors as a substitute which can eventually provide higher sensitivities, lower cost, as well as smaller size and higher levels of integration compared to the conventional QCM and SAW resonant sensors. MEMS resonators have received a lot of attention over the past decade. As a result, a wide variety of microscale electromechanical resonators have been considered for sensory applications lately [5,6]. The majority of these devices utilize piezoelectric [7] or electrostatic (capacitive) [8] electromechanical transduction with both methods having their respective advantages and disadvantages. Piezoelectric transduction requires integration of piezoelectric thin films and metallic electrodes with the micromechanical elements. Such integration usually results in a drastic decrease in the resonator quality factor and is associated with material characterization and quality control issues. Electrostatic excitation on the other hand suffers from relatively small actuation forces available from electrostatic actuators and the need for very thin transduction gaps complicating fabrication of such devices. Furthermore, narrow transduction gaps are also very vulnerable to accumulation of particulate and/or other contaminants in environmental sensing application.

Thermally actuated MEMS silicon resonators have demonstrated high levels of robustness and reliability for sensory applications when direct contact with the surrounding environment is required [9,10]. Thermal actuation is a well known mechanism that can be implemented conveniently at microscale without any fabrication challenges or the need for material integration. Thermally actuated micromechanical resonant devices with frequencies in the hundreds of kHz to a few MHz have been utilized for chemical [9] or physical [10] sensory applications. This work presents another potential sensory application for thermally actuated MEMS resonators.

II. RESONATOR DESCRIPTION

Figure 1 shows the COMSOL modal analysis of the resonator structures utilized in this work. The resonator consists of a central square mass and four support beams on its four corners. Thermal actuation of this resonator occurs by passing a combination of a DC and an AC current through the narrow thermal actuator beams embedded in the supports. This results in a fluctuating ohmic loss in the structure. Due to their higher resistance, most of the heat is generated in the narrow actuator beams. The AC force generated in the actuator beams as the result of the fluctuating temperature and therefore fluctuating thermal stress in the beams can actuate the resonator in its in-plane resonance mode in which the central mass vibrates back and forth as shown in Figure 1.

This work was supported by National Science Foundation (NSF) under grant #0800961.
The alternating stress in the beams leads to fluctuations in their electrical resistance (due to the piezoresistive effect). When biased with a DC voltage, such resistance fluctuations modulate the current passing through the structure resulting in an AC current component known as the motional current.

III. FABRICATION

A single mask process was used to fabricate the resonators on low resistivity SOI substrates with different device layer and buried oxide (BOX) thicknesses. Figure 2 shows the fabrication process steps that include carving the structures into the SOI device layer (deep reactive ion etching) followed by undercut of the structures by etching the underlying buried oxide (BOX) layer in a concentrated hydrofluoric acid (HF) solution. After the resonators were released and dried, in order to form an absorbent layer on their sensing plates, they were dipped in a dilute solution of 1813 photoresist in acetone (one drop in 50cc of acetone). The excess solution was removed from the resonator chip by spinning at 3000rpm for 30 seconds followed by a hard bake step to polymerize and dry the polymer coating. As a result a thin film of polymer was left on the structures. Figures 3a and 3b show the SEM view of a fabricated 15 µm thick, 2.58 MHz resonator and a 20µm thick, 3.82MHz resonator respectively. Figure 3b shows the resonator after coating with the thin polymer layer.
IV. MEASUREMENT SETUP AND RESULTS

Figure 4 shows the camera picture of the test setup used in this work. The resonators were placed under a sealed bell-jar connected to a vacuum pump with a nozzle placed on top of the resonator sample to suck in the outside air and direct it towards the surface of the devices. The utilization of the vacuum pump together with the sealed bell jar enables a flow of the outside air to be directed more precisely to the surface of the resonator under test. Having a flow of the air sample passing over the resonator surfaces maximizes the contact between potential molecules of interest in the air sample and the resonator surface, hence, maximizing the absorption probability. In this manner, the absorption rate and amount of absorbed molecules for a certain concentration increases dramatically. A particulate filter was placed at the air input to avoid deposition of air-borne particles on the devices causing a wrong response due to the added particle masses. The fabricated resonators were operated in a one-port configuration with the thin actuator beams of the structures acting simultaneously as both thermal actuators and piezoresistive sensors.

Figure 5 shows the change in the measured frequency response for the resonator shown in Fig. 3b upon exposure to gasoline vapor emitted from an open gasoline surface at room temperature.

As can be seen in Fig. 6, upon exposure, the resonance frequency reduces as a decaying exponential over time with a time constant of ~4min. Restoration of the resonant frequency to its initial value happens very quickly as soon as the source is removed (Fig.6).

Figure 7 shows the frequency of the same resonator (after stabilization) for different distances of the chamber input from the gasoline surface. As expected, the frequency shift drops sharply as the source is moved further away.

Fig. 4. The test setup used to characterize the resonator sensory behavior. The resonator is placed in a sealed bell-jar with a micro-jet positioned over the resonator bringing the air sample in contact with the resonator surface due to the pressure difference. This pressure difference is caused by the vacuum pump sucking the air.

Fig. 5. Measured frequency responses for the 3.82MHz resonator of Fig. 3b after different exposure times during gasoline absorption.

Fig. 6. Change in the measured resonance frequency for the 3.82MHz resonator of Fig. 3b as a function of the cumulative exposure time showing an overall frequency shift of ~760Hz (198 ppm). The source was removed after 20 min. The return to a frequency very close to the initial frequency is almost instantaneous.

Fig. 7. Change in the measured resonance frequency for the 3.82MHz resonator of Fig. 3b as a function of the distance of the nozzle from the gasoline surface. As expected, the frequency shift drops sharply as the source is moved further away.
The mass sensitivity of the resonators can be calculated based on their effective mass as follows:

\[
\frac{1}{2\pi} \sqrt{\frac{k}{m}} \Rightarrow \frac{\partial f}{\partial m} = -\frac{f}{2m}
\]

where \(k\), \(m\), and \(f\) are the effective stiffness, effective mass and resonant frequency of the resonator respectively.

Table 1 summarizes the measurement results obtained for different resonators. Since all devices have similar coatings the calculated equivalent absorbed gasoline thickness for different devices is similar.

Knowing the dimensions and therefore the mass of the resonators, the mass of the deposited gasoline \(\partial m\) was estimated from the measured frequency shifts. Having the mass of the deposited gasoline, the volume and consequently the thickness of the absorbed gasoline (assuming a uniform distribution over all resonator surfaces) can be calculated by knowing the gasoline density.

### Table 1

**Summary of Measurement Results Obtained from Different Resonators and the Calculated Equivalent Absorbed Gasoline Thickness Values.**

<table>
<thead>
<tr>
<th>Dimensions (μm)</th>
<th>Actuator Tilt</th>
<th>Bias Current (mA)</th>
<th>Freq. (kHz)</th>
<th>Q. Factor</th>
<th>Freq. Shift (Hz) ppm</th>
<th>Equivalent Gasoline Thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>280 / 280 / 20</td>
<td>15 / 3</td>
<td>45</td>
<td>19.8</td>
<td>3827.6987</td>
<td>2004 -757</td>
<td>12.0</td>
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<td>200 / 200 / 15</td>
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<td>20.1</td>
<td>2582.5274</td>
<td>1370 -486</td>
<td>10.0</td>
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<tr>
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<td>19</td>
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<td>9.3</td>
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<tr>
<td>280 / 280 / 15</td>
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<td>20.4</td>
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<tr>
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<td>0</td>
<td>19</td>
<td>706.5243</td>
<td>1820 -149</td>
<td>11.4</td>
</tr>
</tbody>
</table>

V. CONCLUSIONS

Thermally actuated in-plane silicon resonators were successfully used for detection of the presence of gasoline vapor in air. The thickness of the deposited gasoline vapor on the surface of sensor was calculated according to the change in the resonant frequency of the resonator. Future work includes further design optimization of the resonators and narrowing down their thickness to achieve higher mass sensitivities. Using more suitable absorbent polymer coatings and maximizing of the absorbing surface area using porous coatings could significantly improve the sensitivity.

ACKNOWLEDGMENT

Authors would like to thank Professor Abdolvand and his research group at Oklahoma State University for their help with silicon deep reactive ion etching.

REFERENCES


