Detection and Mass Measurement of Individual Air-Borne Particles Using High Frequency Micromechanical Resonators

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Abstract— This work demonstrates detection and mass measurement of single micro/nanoscale air-borne particles using thermally actuated high frequency micromechanical resonators. Single crystalline silicon dog-bone resonators with resonant frequencies in the tens of MHz range were fabricated using a single mask process on SOI substrates. The mass sensitivities of the resonators were characterized using artificially generated airborne particles and mass sensitivities as high as 1.6kHz/pg were demonstrated. Due to the high mass sensitivities, the shift in the resonator frequencies caused by every individual particle is distinguishable. The measured mass sensitivities are in good agreement with the calculated values based on the resonator physical dimensions.

I. INTRODUCTION

Measurement of concentration and size distribution of atmospheric micro/nanoscale particles is of great interest to environmental scientists. Such particles play important roles in air quality, human health, radiation balance of the earth (climate change), and stratospheric ozone depletion [1]. Furthermore, monitoring particle counts in highly controlled environments and measurement of the number and size of individual particles in gaseous streams is an essential requirement for cleanrooms, clean benches, filter facilities in operation rooms, filling facilities in the pharmaceutical industry and separation efficiency tests of filters. Existing versions of instruments and sensors for such measurements are either based on optical measurement techniques such as nephelometry [2] and polychromatic LED techniques [3], Scanning Electron Microscopy (SEM) [4], or conventional resonant mass sensors such as Surface Acoustic Wave (SAW) resonators [5,6] resulting in sophisticated, immense and costly instruments that in some cases do not provide the desired sensitivity level [5-7]. MEMS resonators, as low-cost highly integrated and ultra-sensitive mass sensors, are expected to enable revolutionary advances in such instruments providing unprecedented capabilities such as direct determination of air-borne particle size and mass (therefore density and even composition) distributions while having significantly lower cost and smaller size. A wide variety of MEMS resonators have been previously used for sensory applications. Most of such devices use piezoelectric [8] or electrostatic (capacitive) [9] electromechanical transduction. Piezoelectric micro-resonators require deposition of piezoelectric and metal thin films generally resulting in lower quality factors and frequency and quality control issues. In addition, it is very hard (if not impossible) to have a uniform mass sensitivity all over their whole sensing plate [10]. Uniform mass sensitivity is a necessity when targeting mass measurement of individual particles. In case of air-gap capacitive resonators, extreme vulnerability of air-gaps to contaminants or particulates makes it practically impossible to use them for particulate sensory applications. Moreover, small actuation forces of electrostatic actuators necessitate deep submicron transduction gaps leading to fabrication challenges, power handling limitations, and excessive squeezed film damping when operating in air. In contrast, electro-thermal actuators are extremely simple to implement requiring only a heating resistance. Thermal actuators have great properties such as large actuation force and low operating voltages. Finally, actuation of in-plane translational resonance modes, providing uniform mass sensitivity, is quite straightforward using thermal actuators. We have previously shown measurement of cumulative mass of aerosol particles using thermally actuated micromechanical resonators [11,12]. This work presents thermally actuated micromechanical resonators capable of mass measurement of single submicron aerosol particles.

II. RESONATOR DESCRIPTION

Figure 1 shows the 3D schematic view of the resonator structures utilized in this work. Such resonators are referred to as I2-Bulk Acoustic Wave Resonators (I2-BARs; also known as dog-bone resonators) [13,14]. Thermal actuation of this resonator occurs by passing a combination of a DC and an AC current between the two pads on the two sides of the structure [14]. This results in a fluctuating ohmic loss in the current.
path. Due to their higher resistance, most of the heat is generated in the thin pillars located in the middle of the structure. The AC force generated in the pillars resulting from the fluctuating temperature and therefore fluctuating thermal stress in the pillars, can actuate the resonator in its in-plane resonance mode. As shown in Fig. 1, in this mode the masses on the two ends of the pillars vibrate back and forth as shown in Figure 1.

At the same time as the resonator vibrates, the alternating tensile and compressive stress in the pillars results in fluctuations in their electrical resistance (due to the piezoresistive effect). Since the resonator is biased with a DC voltage, this results in fluctuations in the DC current passing through the resonator (output signal) that represents the vibration amplitude of the resonator.

III. FABRICATION AND MEASUREMENT RESULTS

The standard single mask SOI process [12] was used to fabricate the resonators on two different low resistivity SOI substrates: 1) a P-type SOI substrate with device layer thickness of 15μm and (BOX) thickness of 5μm, 2) an N-type SOI substrate with device layer thickness of 5μm and buffer oxide (BOX) thickness of 5μm. Figure 2 shows the SEM view of two of the fabricated I²-BARs which were used in our experiments. For the bottom device the actuator beams were thinned down in order to minimize resonator power consumption. This was done by performing a number of consecutive thermal oxidation and oxide removal steps after the devices were released. At the same time, the resonator thicknesses were thinned down as well.

In order to characterize mass sensitivity of the resonators, the silicon chip containing the resonators was placed on a printed circuit board (PCB) containing the required resistors and capacitors to apply DC bias and AC actuation currents to the resonators. Connections to the resonators were provided by wedge-bonded aluminum wires. The PCB was then embedded in a custom made system comprised of a sealed chamber, an aerosol particle generator, and an alignment apparatus. The schematic diagram and camera picture of the test setup is shown in figure 3.

In the aerosol particle generator, a flow of liquid solution of methylene blue in ethanol generated by a micro syringe pump is first turned into small droplets (atomized) by a nitrogen flow. The droplets are passed through a Kr-85 bipolar diffusion charger that neutralizes most of the charge left on the droplets as a result of atomization and establishes a charge distribution close to Boltzman distribution for the droplets (mostly neutral, some+1q, less ±2q, etc.). In the meanwhile the solvent in the drops is evaporated and the dried aerosol is injected into a differential mobility analyzer that separates the particles using an electrostatic field (based on their mass and electric charge) allowing only particles with specific diameter and charge to pass through it. The electric field and flows were regulated to permit selection of
particles having a diameter close to 1μm. Particles coming out of the particle generator are then directed into the low pressure (50-100 Torr) sealed chamber. In the chamber, the PCB is horizontally placed on a micro-positioning stage, with an alignment apparatus sitting under it. The alignment apparatus consists of a microscope with one of its objective lenses replaced by the nozzle carrying the flow of particles from the generator. Once the microscope lens is aligned directly on top of the specific resonator to be characterized, the head is turned so that the nozzle points directly towards the resonating device. Therefore particles coming out of the particle generator are deposited on the resonator under test. Resonators with different dimensions were exposed to the flow of particles for several consecutive intervals of a few minutes each. After each interval, the resonator characteristics were measured and recorded.

Figure 4 shows the resonant frequency of the 61MHz and 20MHz resonators of Fig. 2 versus the overall exposure time after each exposure step. It is clear that reduction of the resonant frequency of the resonator is quantized and occurs in steps that are multiples of ~800Hz for the 60 MHz and ~900Hz for the 20 MHz resonator. This makes the effect of every single particle on the resonance frequency distinguishable.

Figure 5 shows the SEM view of the same resonators in Figure 2 after 25 and 24 minutes of exposure showing exactly 5 and 7 spherical particles of ~1µm diameter deposited on their two sensing plates. This is in complete agreement with the step by step frequency shifts shown in Fig. 4. Overall 5 particles and 7 particles have been deposited on the 61MHz and 20MHz resonator respectively.

The mass sensitivity of the resonators can be theoretically calculated as follows:

$$f = \frac{1}{2\pi} \sqrt{\frac{k}{m}} \Rightarrow \frac{\Delta f}{f} = -\frac{m}{2m}$$

(1)

Figure 3. Aerosol particle generator and accelerator used to characterize the resonator mass sensitivities. The aerosol jet was positioned over the sensor using a modified microscope with an integrated nozzle and a micro-positioning stage.
where $k$, $m$, and $f$ are the effective stiffness, effective mass and resonant frequency of the resonator respectively.

Knowing the dimensions and therefore the effective mass of the resonators, the overall mass of the deposited particles ($\delta m$) were estimated from the measured frequency shifts ($\delta f$). The overall mass of the particles was also calculated using the number of the deposited particles given by SEM inspections of Fig. 5. The overall particle mass was then calculated using:

$$\Delta m = N \rho \pi d^3 / 6$$

where $N$ is the number of particles, $\rho$ is the density of the material the particles are made of, and $d$ is the particle diameter. Table 1 summarizes the measurement results for the tested resonators and compares the experimental frequency shift caused by the added mass of the particles (~0.65pg per particle) with the expected frequency shift according to the mass of the resonator and Equation 1, showing a good agreement between the two.

IV. CONCLUSIONS AND FUTURE WORK

Mass of individual artificially generated air-borne microparticles was successfully measured using high frequency thermally actuated in-plane silicon resonators. Mass sensitivities as high as 1.6KHz/pg were measured for the resonators, that are in good agreement with the theoretically calculated mass sensitivities. According to the Allen variance method calculations [15], when embedded in a low noise oscillator configuration, very small frequency resolutions should be achievable for the demonstrated resonators allowing detection of single particles as small as 10nm in diameter. Furthermore, orders of magnitude higher mass sensitivities are achievable by further reducing the thickness and horizontal dimensions of the resonators.

Future work includes further design optimization and miniaturization of the resonator dimensions to achieve higher mass sensitivity. Using multi-layer two port configurations is also among other future directions.
### Table I

**SUMMARY OF MEASUREMENT RESULTS OBTAINED FROM THE TWO RESONATORS IN FIG 3 AND COMPARISON OF THE MEASURED MASS SENSITIVITY WITH THE THEORETICALLY PREDICTED MASS SENSITIVITY.**

<table>
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<th>Q factor</th>
<th>Freq (MHz)</th>
<th>Time (min)</th>
<th>Freq. Shift (Hz)</th>
<th>No. of particles detected</th>
<th>Overall Mass from freq. shift (pg)</th>
<th>Overall Mass from Microscope count (pg)</th>
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**REFERENCES**


