Frequency-tuning for control of parametrically resonant mass sensors

Wenhua Zhang[a)] and Kimberly L. Turner
Department of Mechanical and Environmental Engineering, University of California, Santa Barbara Engineering II, R2355, Santa Barbara, California 93106-5070

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Parametric resonance based mass sensing leads to increased sensitivity over other resonant methods. In this work, we present a frequency-tuning approach to measure mass change in an ultra-sensitive mass sensor. This scheme drives the oscillator using an electrical signal with fixed frequency and tunes the parametric resonance frequency to match the driving frequency by feeding back a dc offset to the sensor. Instead of monitoring frequency shift of the oscillation in a micro-oscillator, mass change in the sensor can be detected by measuring the shift in dc offset, making the sensor amenable to closed-loop control. This scheme and the prototype mass sensor with a mass of ~30 ng and a first mode harmonic resonance frequency of ~83 kHz has been built and experimentally characterized. Good correlation between parametric resonance frequency and dc offset has been shown and the noise floor is less than 1 pg. The sensor is used to track water content variation in a humidity-testing chamber and shows increased efficiency over open loop operation. © *2005 American Vacuum Society.* [DOI: 10.1116/1.1924717]

I. INTRODUCTION

As micro/nanofabrication technology is rapidly evolving, resonance based mass sensors have achieved significant success in improving mass sensitivity in the last few years, benefiting both chemical and biological sensing applications.[1–5] In 1995, T. Thundat *et al.* were able to detect a 31 picogram (pg) mass change using a microcantilever with resonance frequency of 25 kHz.[1] In 2003, Lavrik and Datskos improved mass sensitivity to the femtogram (fg) level using a nanoscale cantilever with resonance frequency of 2.26 MHz at atmospheric pressure.[3] In 2004, B. Ilic *et al.* pushed the mass sensing limit to the attogram (ag) level with a nanoscale cantilever testing at 3×10^{-6} Torr.[5]

To improve the sensitivity of resonance mode mass sensors, much effort has been put in shrinking the size and raising the resonance frequency of the sensors. Combining micro/nanofabrication and focused ion beam technology, nanoscale microbeams with resonance frequencies >1 MHz can be fabricated.[3] However, further improvement becomes difficult as the sensor size enters the nanoscale. Sensing also becomes more challenging at these size scales. New materials, such as nanowires and nanotubes, and new technologies need to be explored.

Most current resonance-mode mass sensors, including chemical and biological sensors, operate in a harmonic resonance mode. In this mode, mass changes are detected by measuring shifts in resonance frequency. Theoretically, any mass change leads to a frequency shift. However, the capability to detect this resonance frequency shift is determined by many factors, including noise and the quality factor Q. Many resonance mode mass sensors achieve very high sensitivity under vacuum with high Q, but in atmospheric pressure environment, the sensitivity can decrease by 1–2 orders of magnitude. Thus traditional resonance mode limits the usefulness in applications, such as air or water quality monitoring.

Some methods have been proposed to increase the sensitivity of resonance-mode mass sensors, such as using feedback control to improve quality factor.[6] In our previous work, we have shown that parametric resonance amplification is an efficient technique to improve the performance of resonance mode mass sensor.[7–9] Damping does not have significant effects on the sensitivity of such sensors, aside from slightly increased power requirements.[8]

In general, parametric resonance can be actuated in a dynamic system with time-varying stiffness or mass.[10] In a noninterdigitated comb-finger driven micro-oscillator,[8,9] electrostatic force between fixed and moving fingers is time dependent. Therefore, as an ac signal is applied, such a system has time varying effective stiffness.[8,10,11] Considering mechanical and electrostatic nonlinearity, the dynamics can be described using the nonlinear Mathieu equation,[8] as a square-rooted ac signal is applied to avoid coupling between harmonic resonance and parametric resonance.[10] The equation has been thoroughly investigated in our earlier work[8] and the normalized form is shown below

$$\frac{d^2x}{d\tau^2}+\alpha\frac{dx}{d\tau}+(\beta+2\delta\cos 2\tau)x+(\delta_3+\delta_3'\cos 2\tau)x^3=0 \quad (1)$$

and

$$\alpha=\frac{2c}{m\omega},\quad \beta=\frac{4(k_1+r_1V_A^2)}{m\omega^2},\quad \delta=\frac{2r_1V_A^2}{m\omega^2},$$

$$\delta_3=\frac{4k_3+4r_3V_A^2}{m\omega^2},\quad \delta_3'=\frac{4r_3V_A^2}{m\omega^2},$$

where m, k_1, and k_3 are the mass, linear, and cubic mechanical stiffness of the oscillator, respectively, c is the damping

[a)]Electronic mail: whzh@engineering.ucsb.edu

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coefficient, r_1 and r_3 are linear and cubic "electrostatic stiffness," and $\tau = \omega t$ is a normalized time.[8]

The dynamics of parametric resonance are quite different from harmonic resonance. There are clear boundaries between resonance regions and nonresonance regions, where the boundaries are determined by driving voltage V_A and driving frequency f, as well as other sensor parameters.[8,10] In the frequency domain, there exists a sharp "jump" in the response (displacement or velocity) at the boundary of a parametric resonance region. The frequency at the boundary depends on the parameters of the sensor and the driving signal, including mass m as shown below[9]

$$f = \frac{1}{2\pi}\sqrt{\frac{4k_1 + 2r_1 V_A^2}{m}}. \tag{2}$$

Thus, mass change can be found by measuring the frequency shift. The sharp "jump" in frequency response makes it possible to detect a very small frequency shift. In addition, damping does not affect the existence of parametric resonance as long as enough energy is input into the system to overcome damping. Therefore, a very sensitive mass sensor can be built based on this technique. In a prototype parametric resonance based mass sensor, the resolution and sensitivity can be 1–2 orders magnitude higher in atmospheric pressure environment than the same sensor working in harmonic resonance mode. Frequency shift of about 2 Hz of 160 kHz can be easily detected.[9] Damping has no significant effect on sensing performance and noise floor.[12]

Traditional resonance-mode mass sensors, including the prototype parametric resonance based sensor in our previous work, measure mass changes based on an open-loop strategy, in which the frequency of driving electrical signal is swept up or down to find the resonance frequency shifts and mass change directly based on the following relationship:

$$\Delta m \approx -2\frac{m}{f_0}\Delta f. \tag{3}$$

In this work, we take advantage of the characteristics of noninterdigitated comb fingers[11] and propose a closed-loop strategy to measure mass changes in a parametric resonance based mass sensor. A dc offset is applied to the sensor as a feedback signal to compensate for the frequency shift at the boundary of parametric resonance region (we will use the term "parametric resonance frequency" throughout the text to denote the frequency at the boundary of parametric resonance region). Therefore, mass changes can be detected by measuring the dc offset feedback. This concept is theoretically analyzed and experimentally verified in a prototype mass sensor based on a noninterdigitated comb-finger driven oscillator. The sensor performance and noise floor is characterized as well.

II. THEORY

The prototype mass sensor is a micro-oscillator fabricated from silicon-on-insulator (SOI) wafer and consists of single crystal bulk silicon with a set of noninterdigitated comb fingers, four supporting springs, and a backbone, which moves in plane when actuated. A scanning electron micrograph (SEM) is shown in Fig. 1. The resonance frequency of the oscillator is about 83 kHz and the mass is about 30 ng (nanogram).

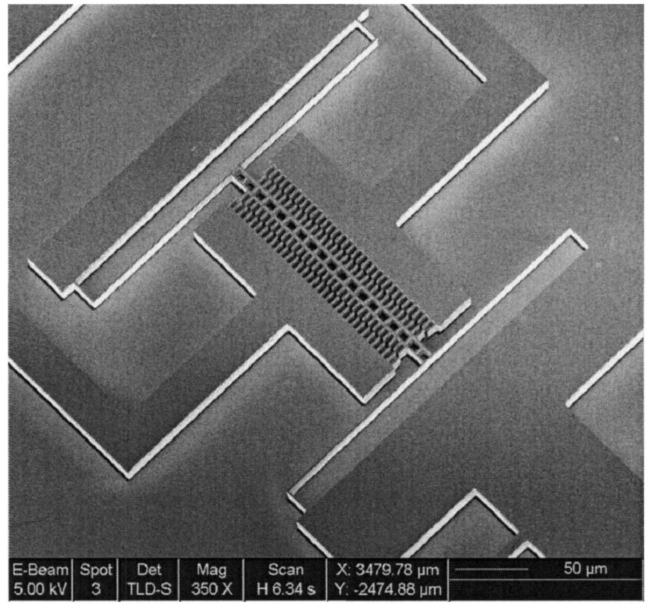

FIG. 1. Prototype mass sensor fabricated from SOI wafer. It is a micro-oscillator with a backbone, four supporting springs, and one set of noninterdigitated comb fingers. The mass is about 30 ng and resonance frequency is about 83 kHz.

Since the micro-oscillator shown in Fig. 1 is driven with noninterdigitated comb fingers, the electrostatic force is position and drive signal dependent.[8,11] The effective stiffness of this dynamic system is a combination of both mechanical recovery force and electrostatic actuation force. Therefore, the resonance frequency and parametric resonance frequency (about twice the natural resonance frequency) can be tuned by changing the dc offset in the ac drive signal.[11] If the frequency of the electrical signal is fixed, by matching parametric resonance frequency with this frequency using dc offset feedback as the mass of the oscillator changes, the mass change can be found by monitoring the dc offset shifts.

This sensing strategy is a closed-loop system with dc offset as the feedback signal, which depends on parametric resonance frequency shifts caused by mass change. Figure 2 schematically shows the block diagram of such a system as well as the open-loop system used by traditional resonance mass sensors.

For noninterdigitated comb fingers, electrostatic force is[8,11]

$$F_e(x,t) = -(r_1 x + r_3 x^3)V^2. \tag{4}$$

As a square rooted ac signal with dc offset feedback $\{V = V_A[1+\cos(\omega t)]^{1/2} + V_{dc}\}$ is applied

$$F_e(x,t) = -(r_1 x + r_3 x^3)\{V_A[1+\cos(\omega t)]^{1/2} + V_{dc}\}^2 \tag{5}$$

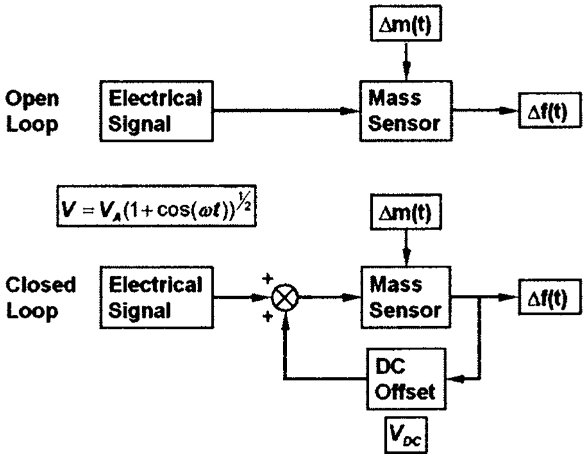

FIG. 2. Open-loop sensing system in traditional mass sensor applications and proposed closed loop system in parametric resonance based mass sensor.

$$= -(r_1x + r_3x^3)\{[V_A^2 + V_{dc}^2] + 2V_AV_{dc}[1+\cos(\omega t)]^{1/2} + V_A^2\cos(\omega t)\}. \quad (6)$$

Using Fourier series expansion

$$F_e(x,t) = -(r_1x + r_3x^3)\{[V_A^2 + V_{dc}^2 + 3.6V_AV_{dc}] + (1.2V_AV_{dc} + V_A^2)\cos(\omega t)\}. \quad (7)$$

If $V_{dc} \ll V_A$, then

$$F_e(x,t) = -(r_1x + r_3x^3)[V_A(V_A + 3.6V_{dc}) + V_A(1.2V_{dc} + V_A)\cos(\omega t)]. \quad (8)$$

Therefore, the governing equation of the oscillator dynamics becomes

$$m\ddot{x} + c\dot{x} + k_1x + k_3x^3 = -(r_1x + r_3x^3)[V_A(V_A + 3.6V_{dc}) + V_A(1.2V_{dc} + V_A)\cos(\omega t)]. \quad (9)$$

After normalization, Eq. (9) becomes

$$\frac{d^2x}{d\tau^2} + \alpha\frac{dx}{d\tau} + (\beta + 2\delta\cos 2\tau)x + (\delta_3 + \delta_3'\cos 2\tau)x^3 = 0, \quad (10)$$

where

$$\alpha = \frac{2c}{m\omega}, \quad \beta = \frac{4(k_1 + r_1V_A^2 + 3.6r_1V_AV_{dc})}{m\omega^2},$$

$$\delta = \frac{2r_1(V_A^2 + 1.2V_AV_{dc})}{m\omega^2},$$

$$\delta_3 = \frac{4(k_3 + r_3V_A^2 + 3.6r_3V_AV_{dc})}{m\omega^2}, \delta_3' = \frac{4r_3(V_A^2 + 1.2V_AV_{dc})}{m\omega^2}.$$

According to the characteristics of parametric resonance, the parametric resonance frequency follows (not considering damping):

$$\beta = 1 \pm \delta. \quad (11)$$

As a result, the following relation holds at the boundaries of the parametric resonance region:

$$\frac{4(k_1 + r_1V_A^2 + 3.6r_1V_AV_{dc})}{m\omega^2} = 1 \pm \frac{2r_1(V_A^2 + 1.2V_AV_{dc})}{m\omega^2}. \quad (12)$$

At the right side boundary where a sharp "jump" in frequency response exists,

$$m\omega^2 = 4(k_1 + r_1V_A^2 + 3.6r_1V_AV_{dc}) - 2r_1(V_A^2 + 1.2V_AV_{dc}) \quad (13)$$

and

$$m = \frac{4k_1 + 2r_1V_A^2 + 12r_1V_AV_{dc}}{\omega^2}. \quad (14)$$

If a fixed square rooted signal with varying dc offset is applied, then

$$dm = \frac{12r_1V_A}{\omega^2}dV_{dc}. \quad (15)$$

We can see that mass change can be detected by measuring dc offset change to actuate parametric resonance. Theoretically, in the small dc offset case ($V_{dc} \ll V_A$), mass change linearly depends on dc offset shift. But this relationship needs to be experimentally verified and noise effects need to be investigated as well before a mass-sensing test, which is verified in the following section.

III. EXPERIMENTS AND RESULTS

As we know from traditional resonance mode mass sensors, including parametric resonance phenomenon based, the mass change is linearly dependent on resonance frequency shift in case of small mass changes, as shown in Eq. (3). In this new closed loop concept, if frequency shift is linearly proportional to dc offset feedback change ($\Delta f \propto \Delta V_{dc}$), then mass change can be found based on Eq. (15). Therefore, the key is the relationship between dc offset change and parametric resonance frequency shift, and we first investigated this relationship experimentally.

The sensor oscillator was excited using a squared rooted ac signal $[V = V_A(1+\cos(\omega t))^{1/2}]$ to determine the parametric resonance frequency. A small dc offset $V_{dc}(V_{dc} \ll V_A)$ was added to the driving signal, which caused the frequency to shift due to the configuration of the noninterdigitated comb fingers,[8,11] as shown in Eq. (13). The relationship between frequency shift and dc offset is shown in Fig. 3. The inset shows this relationship in small scale. A 1 mV dc offset corresponds to a frequency shift of 0.23 Hz.

We also characterized the noise floor in the closed-loop sensor operation. Dc offset feedback at certain fixed conditions (temperature, pressure, mass, stiffness, and driving signal parameters) was measured multiple times at atmospheric pressure to study the fundamental sensor noise floor. The dc fluctuation over time is shown in Fig. 4. The noise floor was ~1 mV and was equivalent to frequency fluctuation of 0.23 Hz according to Fig. 3. This was approximately in the same level as in the open-loop system (0.7–0.8 Hz).[9,12] There was

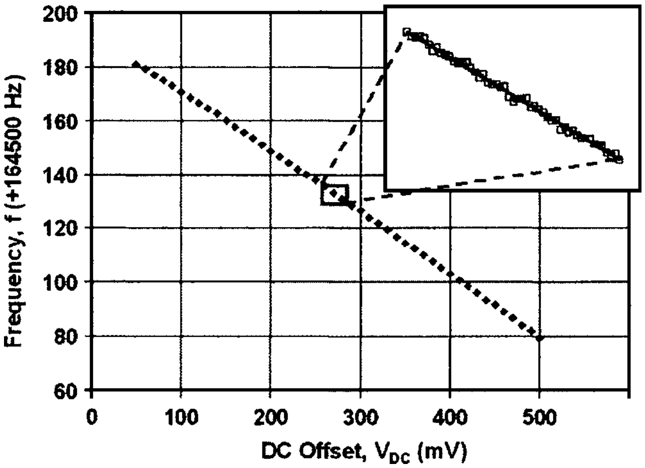

FIG. 3. Linear relationship between dc offset V_{dc}, and parametric resonance frequency f. The inset shows this relationship in small scale.

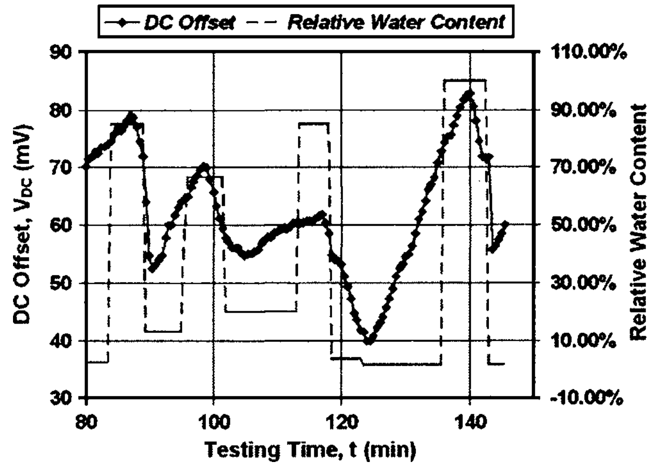

FIG. 5. Dc offset changes with adjustments in relative water content in the testing chamber under atmospheric pressure. The corresponding relative water content changes are also shown.

no significant effect of dc offset on the sensor noise floor, which was equivalent to a mass change of less than 1 pg. Brownian motion noise was the major contribution to the sensor noise floor.[12]

The sensor was tested in a testing chamber with adjustable gas pressure and composition. To minimize environmental effects on the sensor, we changed the mass of the sensor by exposing it to nitrogen with varying water content and measured dc offset simultaneously. As the water content was varied, the mass of the sensor changed. By measuring the dc offset necessary to compensate the frequency shift caused by mass change and keeping parametric resonance frequency at a fixed value, the mass change in the sensor was tracked. Figure 5 shows dc offset changes as relative water content was adjusted in the testing chamber at atmospheric pressure. The corresponding relative water content is also shown in Fig. 5. The millivolt levels of dc offset were easily resolved. According to Eq. (15) and the characterization of the relationship between the frequency shift and the mass change in our earlier work,[9] the mass sensitivity is estimated to be at sub picogram mass change levels.

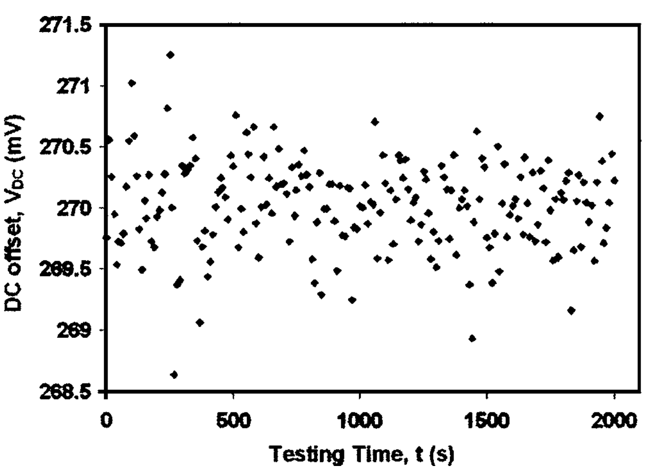

FIG. 4. Noise floor of the prototype mass sensor in closed-loop system. The standard deviation is about 1 mV and is equivalent to a mass change of less than 1 pg.

As shown in Fig. 5, as relative water content changed, dc offset feedback drifted accordingly. However, there was a visible delay between them. The reason was the large volume of the testing chamber (about 200 cm^3). The relative water content shown in Fig. 5 was not the real-time water content in the chamber. Instead, it was calculated based on the flow rates of dry and wet nitrogen gases flowing in to the chamber. Each time as the flow rate changed, it required a few minutes for the actual water content in the testing chamber to change and affect the sensor mass. A smaller chamber is necessary for the sensor to work efficiently.

IV. CONCLUSIONS

In parametric resonance mass sensing, the sensitivity can be improved more than one order of magnitude at atmospheric pressure environment than in traditional harmonic resonance based sensors. Damping does not affect the sensor noise floor.

Based on the characteristics of noninterdigitated comb fingers, a closed-loop sensing strategy is proposed. The functionality and efficiency is experimentally characterized and verified in a sensing test by tracking water content change in a testing chamber. There is no significant effect of dc offset on the sensor noise floor. The prototype mass sensor with mass about 30 ng and resonance frequency about 83 kHz is estimated to be able to measure a mass change of less than 1 pg.

ACKNOWLEDGMENT

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