VACUUM ENCAPSULATED RESONATORS FOR HUMIDITY MEASUREMENT

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ABSTRACT

This paper demonstrates using charge-biased vacuum encapsulated single-anchored double-ended tuning fork (DETF) resonators for humidity sensing. We charge-bias the DETF resonators and measure the change in the stored charge by observing the resonant frequency. Environmental humidity modifies the discharge characteristic of the resonator by varying the resistance between the bias bondpad and the surface silicon dioxide. Using this variation, we demonstrate a sensor that can determine relative humidity from <1 % to 60% with an absolute measurement uncertainty of less than $\pm 0.8\%$ RH.

INTRODUCTION

The resonant frequency of unencapsulated MEMS resonators is sensitive to environmental variables such as ambient temperature, pressure, and humidity, making direct sensing of any single environmental variable difficult. Li, *et al.* observed changes in the discharge characteristics of a contour mode disk resonator in vacuum and air [1]. Additionally, operating the resonator in atmosphere leads to a significant reduction in the quality factor of the device. Siwak, *et al.* used a cantilever resonator coated with pentacene to detect IPA and ethanol vapors in nitrogen. The resonant frequency shifted due to absorption, which caused a change in mass. When these devices were operated in air, the resonators possessed a quality factor of less than 80 [2]. In order to improve the quality factor of their resonant mass sensor, Berg, *et al.*, placed a fluidic channel inside their resonator and then operated their resonator in a gaseous environment or vacuum [3].

In this paper, we use perturbation of the charge decay of a charge biased, high quality factor ($Q \sim 10,000 - 15,000$), vacuum encapsulated resonator to measure the relative humidity of the environment. Humidity modifies the surface resistance of the unencapsulated bias bondpad-oxide interface. This changes the discharge characteristics of the resonator. We track the charge stored on the resonator by observing the electrostatic spring softening term.

THEORY

Device Description

The fabrication process for these hermetically encapsulated, temperature compensated resonators has been discussed by Melamud, *et al.* [4]. The resonators have a beam length of 400 μm and width of 8 μm . The buried oxide layer is 2 μm thick and the device layer has a resistivity of 0.3-0.7 $\Omega\text{-cm}$. The aluminum bond pads are 100 $\mu\text{m} \times 100 \mu\text{m}$. They are on top of 3.2 - 3.3 μm of LTO deposited at 450° C. The oxide between the cap and the device layer is 2.6 μm of TEOS that consisted of 4 depositions. After each deposition, the wafers are annealed at 1100° C for 1 hour.

Device Operation

Under small resonant amplitude, the resonant frequency of the resonator is given by

$$f \approx f_0 \left(1 - \frac{k_e}{2k_m} \right) \quad (1)$$

where k_m is the mechanical spring constant and k_e is the electrostatic spring softening term. The charge relaxation time of a

Figure 1: Resonant frequency versus charge stored on the resonator. The charge corresponds to bias voltage of 0 – 50 V.

uniform conductor is given by

$$\tau_e = \frac{\epsilon}{\sigma} \quad (2)$$

where ϵ is the dielectric constant of the material and σ is the conductivity of the material. For our resonators, the relaxation time is $3.1 - 7.25 \cdot 10^{-13} \text{ s}$. This is significantly less than the period of oscillation ($\sim 3 \mu\text{s}$) so we can assume equipotential on the resonator beam. Assuming also the voltage on the sense and drive is much less than the effective voltage on the resonator, the electrostatic spring softening term, k_e , is given by

$$k_e = \frac{2A\epsilon_0 Q_{er}^2}{g(gC_3 + 2A\epsilon_0)^2} + \frac{A\epsilon_0 C_3 V_d^2 (gC_3 + A\epsilon_0)}{2g(gC_3 + 2A\epsilon_0)^2} \quad (3)$$

where g is the gap between the resonant beam and the electrodes, C_3 is the parasitic capacitance between the resonator and ground, Q_{er} is the charge stored on the resonator, and A is the area of the electrode. However, since the first term dominates for our devices, the spring softening term reduces to

$$k_e \approx \frac{2A\epsilon_0 Q_{er}^2}{g(gC_3 + 2A\epsilon_0)^2} = \frac{2A\epsilon_0 V_{er}^2}{g^3} \quad (4)$$

where V_{er} is the effective voltage on the floating electrode resonator. Figure 1 shows the resonant frequency versus the charge stored on the DETF. As the charge decays, the resonant frequency will increase because electrostatic spring softening decreases. Additionally, the sense current produced at resonance is given by

$$I_s = \frac{A\epsilon_0 V_d}{g^2} \frac{Q\omega_o}{k} \frac{A\epsilon_0}{g^2} \frac{Q_e^2}{(C_3 + 2A\epsilon_0/g)^2} = \frac{A\epsilon_0}{g^2} V_d \frac{Q\omega_o}{k} \frac{A\epsilon_0}{g^2} V_{er}^2 \quad (5)$$

where Q is the mechanical quality factor, ω_o is the resonant frequency, and V_d is the amplitude of the drive voltage. For both the current and the spring softening, the equations are similar to the case of dc biasing the resonator. Additionally, both the amplitude of the signal and the resonant frequency depend on the stored charge squared.

Leakage Mechanisms

Leakage changes the amount of charge stored on the resonator. The primary leakage mechanisms for a floating electrode resonator are surface conduction from the aluminum

bond pads and conduction through the internal oxide layers (Fig. 2). Surface conduction depends on the relative humidity of the external environment.

Expanding on Ho, *et al.*, Fig. 3 shows a circuit model for the leakage from the resonator. The bias electrode voltage is modeled by

$$\frac{\partial V}{\partial t} = \frac{1}{R_s C_s} \frac{\partial^2 V}{\partial x^2} - \frac{V}{R_b C_s}, \quad (6)$$

$$-C_d \frac{\partial V_d}{\partial t} = \frac{V_d}{R_d} - \frac{S}{R_s} \frac{\partial V}{\partial x} \Big|_{x=0}, \quad (7)$$

$$V_d = V(x=0) = \frac{Q_{er}}{2A\epsilon_0/g + C_s} \quad (8)$$

where R_s is the surface resistance, C_s is the capacitance between the surface and ground, R_b is the resistance between surface and ground, C_d is the device capacitance, R_d is the resistance between the device and ground, V_d is the voltage on the device, and S is the bond pad perimeter [5].

Equations 6, 7 and 8 were numerically solved in MatLab using the finite difference method. The device charge decay was fitted to a single exponential function to determine the charge decay time constant, τ . Figure 4 shows the case where the resistivity of R_d equals the resistivity of R_b . Figure 5 uses the experimentally determined value for R_d .

The simulations show that at high surface resistivity ($R_s \rightarrow \infty$), $\tau \rightarrow R_d C_d$ because surface conduction becomes negligible. At low surface resistivity ($R_s \rightarrow 0$), $\tau \propto R_s$ because the leakage is similar to constant dose diffusion or a distributed RC line. From these simulations, we can determine the change in surface resistance from the change in the charge decay time constant.

Surface conduction on insulators, R_s , is determined by electrons hopping between adsorbed spots of water on the surface. Using B.E.T. theory for surface adsorption, the surface coverage ratio can be determined. From that, the surface resistance of silicon dioxide is given by

$$R_s = R_{s0} \exp \left(\alpha \left(\frac{1-h}{Ch} \right)^{1/2} \right), \quad (9)$$

$$C \approx \exp \left(\frac{E_1 - E_L}{RT} \right) \quad (10)$$

where h is the relative humidity (RH), R_s is the resistance at 100% RH, α is a constant that depends on the electron mass, diameter of adsorbed spots and the energy of electrons on the surface, E_1 is the heat of adsorption for the 1st adsorbed water monolayer and E_L is the heat of adsorption for the subsequent monolayers [6].

At low electric fields, through-oxide current in low temperature oxide is produced by charge hopping between states in the forbidden zone. For this hopping conduction, the resistivity for

$$R_d \text{ is given by } \rho = \rho_0 \exp \left(\frac{E}{kT} \right) \quad (11)$$

where E is the activation energy, k is the Boltzmann constant and T is the temperature [7].

Experimental setup

A resonator chip is mounted on a PCB and placed in a custom humidity and temperature controlled chamber. The relative humidity is measured using a HIH-4021 humidity sensor, which has an accuracy of 3% RH. By adjusting the flow of wet and dry nitrogen, the chamber can reach RH from <1% to >99%. To change the temperature of the chip, a peltier thermoelectric

Figure 2: DETF cross-section showing primary leakage paths and locations of parasitic capacitances and resistances. Figure is not to scale.

Figure 3: Equivalent circuit for the discharge of the DETF.

Figure 4: Finite difference model showing τ for different R_s and ρ . Assumes $\rho_b = \rho_d = \rho$.

Figure 5: Finite difference model showing τ for different R_s and ρ . Assumes $\rho_b = \rho$ and $R_d = 7 \cdot 10^{13} \Omega$.

Figure 6: Measurement test setup (Block Diagram).

heater/cooler is placed between a heat sink and the PCB. The temperature is monitored using National Semiconductor LM34CZ (accuracy = 0.56°C at 25°C) and a feedback network is used to control the temperature.

The resonator is biased through the anchor for 10 seconds using a relay inside the humidity box and a HP 3631A voltage supply outside of the box. An AC signal is applied to the two outside drive electrodes and the signal is sensed from the inside sense electrode. While performing a humidity sweep, the humidity is allowed to stabilize for 10 minutes before performing the measurement. Additionally, we wait five minutes after complete decay before performing another measurement. This is to ensure that the initial charge distribution on the surface oxide is the same for all measurements. The signal from the resonator is amplified with a transimpedance amplifier. The resonant frequency, amplitude, and quality factor of the resonator were measured in approximately 7 second intervals using a HP 3577a network analyzer (Fig. 6). A measurement span of 60kHz is used to ensure successful peak tracking during the decay, and the peak frequency is measured with a resolution of 150 Hz.

RESULTS

The charge decay constant τ is defined as the time required for electrostatic spring softening to decrease by a factor of e . To determine τ , the data is fit to a single exponential using the Curve Fitting Toolbox in MatLab. Fig. 7 shows a typical change in resonant frequency as the charge decays over time and the single exponential fitting function. As seen in the analytical model, as the charge decays, spring softening reduces, and the resonant frequency increases. After 300s, the signal level becomes equal to the noise level.

For one resonator, three trials were performed by ramping the relative humidity from $<1\%$ to $>99\%$ with a step size of 10% RH. Figure 8 shows the fitted τ for the different relative humidities. For relative humidities above 60%, the decay occurs in less than 7 seconds, which is too fast to be captured by the current test setup. Figure 9 shows the absolute measurement uncertainty for these measurements. This uncertainty was calculated by multiplying the standard deviation with the sensitivity of the measurements. For all measured relative humidities, the measurement uncertainty is less than the 3% relative humidity accuracy for the humidity sensor that is used to control the humidity in the box. Additionally, removing the single outlier from the 50% RH values, the relative humidity error for 50% becomes 0.754% RH. For that case, the relativity humidity error is less than 0.81%.

Figure 7: Change in resonant frequency as charge decays at $<1\%$ RH. Dots are the data points and the line is the fitted function using a single exponential fit.

Figure 8: Charge decay for relative humidities from $<1\%$ to $>99\%$ RH. Test performed three times using the same resonator.

Figure 9: Absolute measurement error for relative humidities from $<1\%$ to $>99\%$ RH. Test performed three times using the same resonator.

Figure 10: Charge decay for <1% relative humidity at 22°C, 28°C, 36°C, 40°C and 46°C. Test performed three times using the same resonator.

Figure 11: Charge decay for relative humidities from <1% to >99% RH. Test performed three times using the same resonator.

Fig. 10 shows τ versus temperature with <1% relative humidity for a resonator. Using MatLab, the average of the three sweeps were fitted to equation 11 and the resulting r-squared value is 0.99. The fitted activation energy is 0.92 eV which is close to the values observed by Belousov, *et al.* for conduction in low temperature silicon dioxide [8]. Therefore, at <1% relative humidity, the dominating charge loss mechanism is hopping conduction through the oxide. Assuming that $\tau = R_d C_d$ at 0% RH, $R_d = 7.14 \cdot 10^{13} \Omega$ at 295 K.

Unfortunately, the low R_d resistance severely limits the sensitivity of the device to humidity because for most of the humidity range this resistance dominates the discharge mechanism. Ho, *et al.*, measured surface resistance of untreated thermal oxide between $10^{20} - 3 \cdot 10^{15} \Omega$ for relative humidities between 0% and 80% and assuming that $R_b = \infty$. Fig. 11 also shows that the device resistance is contributing to the decay of the charge. If surface resistance was dominating, the line in the figure would be linear because of equation 9.

To improve the device performance, the resistance between device and ground (R_d) must be significantly larger than the surface resistance. This could be accomplished by growing thermal oxide before depositing LTO or TEOS. Additionally, another surface material could be used that possesses a lower surface resistance.

CONCLUSIONS

We have demonstrated that charge-biased microshell-encapsulated single-anchored double-ended tuning fork (DETF)

resonators can be used for precision humidity sensing. These sensors electrically sense the change in resonant frequency as the charge decays from the resonator through an external metal-oxide interface. Additionally, vacuum encapsulation protects the resonator from the environment and we achieve a quality factor of 10,000-15,000.

Currently, we are fabricating devices that use a combination of fusion wafer bonding and epitaxial reactor sealing [9]. These devices should have significantly higher R_d because the oxide between the device and cap will be thermal oxide instead of TEOS. We are examining how to perform humidity sweeps at different temperatures by using a recirculator to heat the nitrogen and the bubbler. Additionally, self assembled monolayers, including HMDS, can be used to change the surface conduction properties.

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