Temperature effects on a high Q FBAR in liquid

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ABSTRACT

In this paper, we present the analysis of temperature effects on a zinc oxide (ZnO)-based film bulk acoustic resonator (FBAR) having a high quality factor (Q) in liquid environments. Q up to 120, an improvement of at least 8× greater than state-of-the-art devices in liquids, is achieved by integrating a microfluidic channel with thickness comparable to the acoustic wavelength in the FBAR. However, the FBAR has a significant temperature sensitivity, which degrades Q and shifts its resonant frequency, resulting in undesirable false-positive/negative responses. To minimize the temperature sensitivity, we analyze sources of temperature effects and characterize FBAR's resonant frequency in a Pierce oscillator. The frequency shift is compensated by tuning the supply voltage of the oscillator, achieving a large tunability of −4300 ppm/V. Measurements demonstrate that Q variation is well controlled within −2.5% per centigrade for a FBAR with a channel thickness of 3.9 μm while the temperature coefficient of oscillation frequency (TCF) reduces from −112 ppm/K to less than 1 ppm/K.

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1. Introduction

For biomolecular detection, label-free methods are attractive and extensively studied since labeling may alter chemical properties of target molecules [1]. However, label-free detection methods, such as Surface Plasma Resonance (SPR) [2] and Quartz Crystal Microbalances (QCM) [3], are significantly less sensitive than labeling methods [1]. Offering the common benefits of micromachining technologies, FBAR is an attractive candidate for implementing a miniature non-labeling biomolecule sensor due to its high resolution and excellent sensitivity [4]. ZnO-based FBAR biosensors reported in recent literature exhibit a mass sensitivity of above 1000 Hz cm−2/ng in air, much higher than that of traditional QCMs [5]. Nevertheless, previous research reveals a significant compromise in mass resolution caused by Q degradation when FBARs are exposed to liquid environments; ~10 ng/cm2 with Q of 15 in water vs. 0.5–1 ng/cm2 with Q of 250 in the air [6,7]. In order to overcome the limitation, we formed a microfluidic channel directly on top of a FBAR to confine the thickness of liquid, which minimizes acoustic energy loss. Q up to 120, approximately 8 times higher than prior art was achieved [8].

Though such implementation shows potential for high resolution and high sensitivity biosensors in liquid environments, the performance is compromised by a significant temperature sensitivity of the FBAR sensor. Q of the sensor is degraded by temperature change which is mainly caused by thermal expansion of materials, acoustic wave velocity and viscosity changes of liquid media. Moreover, temperature drifts the resonant frequency of the FBAR as large as −60 ppm/K [9]. Since the FBAR biosensor detects a mass change by measuring a resonant frequency shift, the sensor may provide false-positive/negative responses if the temperature sensitivity is not appropriately compensated or otherwise corrected.

In this paper, we analyze the sources of Q degradation of FBAR and characterize the FBAR in a Pierce oscillator to compensate its frequency shift by tuning the supply voltage. The paper is organized as follow: In Section 2, we present the schematic and fabrication process of the high Q FBAR biosensor. In Section 3, we describe the mechanism of high Q in liquid for the designed FBAR and present the analysis of temperature effects on Q and frequency shift. Approaches to achieve thermally insensitive Q of FBAR and compensation mechanism for frequency shift are also described. Measurement results and discussions are presented in Section 4. Finally, concluding remarks are presented in Section 5.

2. High Q FBAR in liquid

The schematic of the FBAR biosensor is shown in Fig. 1(a). It consists of a piezoelectric layer (ZnO) sandwiched by two electrodes. The FBAR is supported by a SiN membrane to isolate acoustic energy leakage to the silicon substrate. A microfluidic channel is formed directly on top of the FBAR to achieve high Q in liquid. Fig. 1(b) illustrates the fabrication process of the FBAR. The fabrication starts with a (100) silicon wafer; 2000 Å thick oxide is first thermally grown on the wafer, followed by a deposition of 6000 Å thick low-stress LPCVD SiN layer. 1600 Å thick Al is evaporated and patterned...
Fig. 1. (a) Schematic of a FBAR integrated with a microfluidic channel, (b) fabrication process: (i) growth of SiO\textsubscript{2} and deposition of low stress SiN by LPCVD; (ii) Al, ZnO and Cr/Au deposition and patterning; (iii) release of the membrane by deep RIE and removal of SiO\textsubscript{2} beneath silicon nitride; (iv) PDMS patterning using a photoresist mask, and (v) PDMS etching and cover glass sealing, and (c) a fabricated FBAR with a microfluidic channel.

on the SiN layer as a bottom electrode, which determines the effective area of the FBAR. ZnO, a piezoelectric layer, is then sputtered and patterned. 300 Å/1500 Å thick Cr/Au is sputtered and patterned by lift-off over the ZnO layer as a top electrode. Au is used as the top electrode due to its excellent conductivity and good affinity for biomolecular binding. Then 5 μm thick PDMS is spin-coated on the FBAR. With a thick photoresist layer as a mask, the PDMS layer is patterned by CF\textsubscript{4}/O\textsubscript{2} reactive ion etching (RIE) to form sidewalls of the microfluidic channel. A cover glass serves as a ceiling of the channel and seals the microfluidic channel. The channel thickness is determined by the thickness of the PDMS layer. Different channel thickness can be obtained by carefully etching the PDMS layer to a desired thickness. The wafer is then etched through from the backside by Deep RIE to release the SiN membrane and to form the channel inlets/outlets. The SiO\textsubscript{2} layer underneath the SiN membrane is removed by buffered oxide etchant to obtain a smooth SiN supporting membrane. Fig. 1(c) shows a fabricated FBAR with a microfluidic channel.
3. Temperature effect

Typical MEMS FBARs have high Q in the air/vacuum ranging from a few hundreds to a few thousands. This is because the acoustic wave generated in the FBAR body is well-trapped by the very large acoustic impedance mismatch between the solid materials and air [10]. However, when the FBAR is immersed in liquid, the solid–liquid interface becomes leaky for the acoustic wave as the impedance mismatch is small, resulting in the degradation of the Q. By confining the liquid to a thickness comparable to the acoustic wavelength, the acoustic energy dissipation in liquid is minimized and hence improves Q of the FBAR. Q in liquid exhibits an oscillatory behavior as the thickness of microfluidic channel changes. Thus, it is possible to achieve high Q FBAR in liquid by optimizing the thickness of the microfluidic channel.

However, the optimized Q could be compromised as it is sensitive to temperature. There are several sources for the temperature sensitivity of Q: (i) thermal expansion of structural materials and the substrate, (ii) acoustic velocity change, and (iii) liquid viscosity change. Thermal expansion becomes a dominant source for Q degradation when the channel suffers from large deformation by thermal expansion. This is because thermal expansion shifts the optimized thickness of the channel and consequently lowers Q as temperature changes [8].

Stabilization of Q could be, obviously, achieved by minimizing channel deformation due to thermal expansion, where channel material matters. If ceiling material of the channel is not chosen properly, i.e., materials having large coefficient of thermal expansion (CTE) and small Young’s modulus, the effect of the thermal expansion is very significant. Thermal expansion in lateral direction is in the order of 0.01 μm/K (assuming CTE of the material is 100 ppm/K and channel width is in the order of 0.1 mm). Consequently, lateral expansion will deform the channel in vertical direction which is in the same order, 0.01 μm/K. To minimize such large deformation due to thermal expansion, the ceiling of channel should be made of a material with high Young’s modulus and small CTE such as glass (70 GPa, 4 ppm/K). In this case, the lateral thermal expansion cannot deform the ceiling in vertical direction as the ceiling is made of a stiff material. Then the channel thickness change is solely from thermal expansion of PDMS sidewalls in the vertical direction, which is very small. The PDMS sidewall is only 3–5 μm in thickness and CTE of PDMS is around 100 ppm/K [11,12], hence produces a channel thickness variation in the order of 0.3 nm/K, which is negligible. For such a structure, Q variation mainly comes from acoustic velocity change and viscosity change.

A six-layer transmission line model is used to analyze Q variation [13]. In the transmission line model, each composite layer of the FBAR is modeled by a section of transmission line with its own characteristic impedance. Q of FBAR could be estimated from the total impedance of the six layers. Acoustic velocities and viscosities at different temperatures are incorporated in the model to calculate the Q at different temperatures. Fig. 2(a) illustrates the Q variation resulted from thermal expansion, acoustic velocity, and viscosity change. Thermal expansion has a negligible effect on Q as discussed before. Viscosity of water decreases as temperature increases, resulting in improving Q of FBAR. On the other hand, acoustic velocity increases as temperature changes to lower Q of FBAR. To some extent these two effects compensate each other. However, the velocity effect is substantially larger than the viscosity effect to degrade Q of FBAR.

In addition to the Q variation, temperature also detrimentally affects resonant frequencies of FBAR. Temperature alters material properties of the FBAR including acoustic velocity, viscosity of liquid, layer thickness and density, all of which affect the series/parallel resonances of FBAR [14]. To characterize the resonant frequency of FBAR and compensate the frequency shift caused by temperature, we configure the FBAR in a Pierce oscillator. The oscillator consists of an amplifier (inverter), two capacitors and a resonator to form an oscillating loop, whose oscillation frequency resides between the series and parallel resonant frequencies of the resonator [15]. As shown in Fig. 3(a), FBAR forms a feedback loop between C1 and C2 with a BJT in common-emitter configuration. R1, R2, R3 and R4 bias the transistor in active operation. C3 shorts R4 at the working frequency to ensure common-emitter operation. Inductor, L, is a RF chock which blocks high frequency signals back to the power supply. To assemble the oscillator, a fabricated FBAR is glued and wire bonded on a PCB. The PCB board is printed by a milling machine (LPKF Laser and Electronics AG) that patterns the plated copper layers on both sides of the board. At the center of the board, locates a BJT, NEC NE68119. Standard SMD components with case 0402 are soldered around on the board. A 50Ω connector is fixed at the edge to connect the oscillator output to a spectrum analyzer via a 50Ω cable. Two wires are soldered on the board to connect to a 3 V DC power supply. Fig. 3(b) shows a photograph of the FBAR-based Pierce oscillator on the PCB. The oscillation frequency is determined by the FBAR and resides in between the series and parallel resonant frequencies of the FBAR. As temperature shifts the resonant frequencies of FBAR, the oscillation frequency is shifted by temperature change. To control the ambient temperature, a radiant heater, Holmes HQH307, is used. A thermocouple is mounted approximately 0.5 mm away from the FBAR, as shown in Fig. 3(c). Temperature uniformity across the FBAR is minimal as the effective area of FBAR is only about 100 μm × 100 μm. The distance between heater and FBAR is adjusted to achieve desired temperature reading from the thermocouple, which has an accuracy of 0.1 K. Measurement data is recorded after the ambient temperature stabilizes for a few minutes to accommodate the large thermal mass of the FBAR die and PCB.

In order to compensate the frequency shift, we tune the supply voltage of the oscillator which changes DC and AC voltage across the FBAR. The DC voltage induces higher stiffness in the ZnO film
Fig. 3. (a) Schematic of a Pierce oscillator with FBAR, (b) assembled FBAR biosensor in the Pierce oscillator, and (c) schematic of ambient temperature control.

(electrical stiffening) [16], which changes the resonance frequency of FBAR and in turn changes the oscillation frequency. On the other hand, the AC voltage regulates the temperature of FBAR as AC voltage generates heat within the resonator that also causes oscillation frequency change. The temperature compensation is achieved when the net frequency drift caused by the two effects sums to be zero, which is expressed by

\[ t_v \Delta V + TCF_{osc} \Delta T = 0 \]  

(1)

where \( TCF_{osc} \) is the temperature coefficient of oscillation frequency, \( t_v \) is the relative frequency tunability of supply voltage, \( \Delta T \) is the temperature change and \( \Delta V \) is the tuning voltage required for the compensation.

4. Results and discussions

\( Q \) of FBAR is first examined as temperature changes. A radiant heater is used to control ambient temperature and a thermocouple is mounted near the FBAR to read the temperature. FBAR chips are placed on a probe station and characterized using Agilent E5071C network analyzer. Impedance of the FBAR is extracted from the S11 spectrum. \( Q \) of FBAR could be calculated by [17]:

\[ Q = f \frac{d\phi}{df} \]  

(2)

where \( d\phi/df \) is the slope of impedance phase vs. frequency curve and \( f \) is the work frequency. S11 is measured after temperature becomes stable for tens of seconds. Five measurements of S11 are recorded for each temperature starting from 25°C. \( Q \) is finally obtained in ADS (Agilent Design System) by using Eq. (2). The measured \( Q \) at different channel thicknesses is normalized and superimposed in Fig. 2(b). The channel thicknesses are from 3.7 µm to 3.9 µm by controlling the PDMS thickness using RIE. The measured \( Q \) shows similar trends to the estimated, approximately 10% \( Q \) decrease by 3°C temperature change. The measurements uncertainties are 5–8% for each measurement. \( Q \) of FBAR is very sensitive to the channel thickness as well as temperature as anticipated.

To evaluate the frequency drift caused by temperature, oscillation frequencies of the FBAR-based oscillator are measured at different temperatures. The measurement also starts with 25°C. In the experiment, temperature stability within 0.1°C is required to have a frequency stability of approximately 10 ppm. The measured data are plotted in Fig. 4; the slope of the line representing the temperature coefficients. The oscillation frequency shows a temperature coefficient of \(-112\) ppm/K. The temperature coefficient is not only determined by resonances of FBAR, but also associated with the energy loss of FBAR, transistor parameters, SMD capacitors in parallel with the FBAR and parasitics from interconnects.

The frequency tunability of the oscillator is characterized by measuring oscillation frequency vs. tuning voltage. While oscillation frequency is measured by the spectrum analyzer, the tuning voltage is read from the DC power supply. A thermocouple is used to monitor ambient temperature. The tunability measurement is performed at 25°C and starts with a supply voltage of 3 V. It is important to keep the temperature near FBAR constant at 25°C. If the temperature increases above the reference (25°C) during the tuning of supply voltage, the frequency tunability of supply voltage will be overestimated. Measured oscillation frequency and the tuning voltage are plotted in Fig. 5(a). Oscillation frequency shows very linear with the tuning voltage, and it demonstrates a large tunability of \(-4300\) ppm/V.

By knowing frequency tunability of supply voltage and temperature coefficient of the oscillator, the tuning voltage for compensation is calculated from Eq. (1). In Fig. 5(b), to compensate a temperature change of 5°C, the supply voltage only needs to be tuned by 0.14 V owing to the large tunability of the oscillator frequency. Fig. 6 demonstrates uncompensated and compensated oscillator frequencies for 5°C change in temperature. The compensated frequencies fluctuate around extrapolated line, which is caused by, we believe, the inaccuracy of DC power supply. Yet the
the tuning technique, the output frequency of the oscillator is kept within 1 ppm/K over the temperature change of 5 °C.

References


Biographies

Xu Zhang received the BS degree in electronic engineering from Tsinghua University, China, in 2006. He is currently a PhD student in the Department of Electrical Engineering at Arizona State University. His research interests include MEMS sensor, MEMS integration with electronics and micropackaging.

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