Design, fabrication and characterization of microelectromechanical resonators for chemical and biological applications

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Design, Fabrication and Characterization of Microelectromechanical Resonators for Chemical and Biological Applications

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SCHOOL OF MECHANICAL AND AEROSPACE ENGINEERING

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Summary

The main objective of this thesis is the performance improvement of microresonators as mass-sensitive biochemical sensors. Resonant microstructures fabricated on silicon substrates with micromachining techniques are mainly investigated in this work, enabling the use of well-established technologies for sensor array integration in the future. In this work, two particular resonator types are designed, fabricated and their mass sensitivities characteristics are studied and compared in both gaseous and liquid environments to be used in biochemical detection application.

The first designed resonator is a single-crystal silicon squared resonator with dielectric filled capacitive excitation mechanism. This resonator is fabricated using the silicon-on-insulator MEMS process. In order to study the mass sensitivity of the fabricated resonator, a polyelectrolyte multilayer (PEM) is used to coat on the resonator surface. The resonator has the resonance frequency of 34.81 MHz and mass sensitivity of $105.4 \mu\text{m}^2/\text{ng}$ and the limit of detection of $1.36 \times 10^{-6} \text{ ng}/\mu\text{m}^2$. Experimental measurements of the mass sensitivities agree with theoretical predictions. The developed sensor shows a high potential to be used in bio detection applications compared to some of the state-of-the-art mass sensors.

The second resonator is piezoelectric AlN resonator with checker-patterned electrode architecture. The mass sensitivity of the developed sensor is analytically derived and confirmed with FEM simulations and experimental results. The results show a high mass sensitivity of $1356.81 \text{ Hz.cm}^2/\text{ng}$ ($175.42 \mu\text{m}^2/\text{ng}$) and limit of detection of $18.7 \text{ ng}/\text{cm}^2$ for 773 MHz resonator. This high mass sensitivity arises from the high resonance frequency which can be independently set from the resonator thickness for this special design. The obtained mass sensitivity of the developed sensor is much higher than state of

the art mass sensors and shows the high potential of the developed piezoelectric transducers for mass sensing applications.

Finally, the Lamé-mode resonator is demonstrated as a biological sensor in liquid environment. Before using the bio entities, the mass sensitivity of the resonator is obtained with sequential deposition of Polyelectrolyte Multilayer (PEM) in air and water. Deposition of PEM is used to characterize the sensor to simple mass deposition. Then, specific protein binding interactions using the well characterized biotin/streptavidin system is performed to verify the performance of the sensor with bio materials. The resonant frequency of the biotin modified resonator is measured to decrease by 315 ppm when exposed to streptavidin solution for 15 minutes with a concentration of higher than 10^{-7} M (Molar), corresponding to an added mass of 3.43 ng on the resonator surface. An additional control is added by exposing a BSA covered device to streptavidin in absence of the attached biotin. No resonance frequency shift is observed in this case, confirming the specificity of the detection. The sensor-to-sensor variability is also measured to be 4.3%. Consequently, the biotin modified developed sensor can be used to observe the biotin-streptavidin interaction without the use of labelling or molecular tags. The developed sensor can be used in a variety of protein-ligand systems in bio sensing application.

Chapter 1:

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1. Introduction

1.1 Motivation

In recent years, the chemical/biochemical sensors has been extensively used in many applications such as detection of explosives, DNA and antibody [1, 2]. Although the chemical/biochemical sensor market growth rate is relatively high, the majority of chemical analyses are still dependent on costly laboratory analyses using bench-top instruments. These are highly sensitive and selective, but the analyses are performed off-line and typically time and labor intensive, thus limiting their applications. Therefore, cost effective, highly sensitive chemical/biochemical sensors with ability of in-place measurement are still in great demand.

From this point of view, micromachined chemical/biochemical sensors, which make use of well-established industrial semiconductor fabrication processes, are one of the most promising candidates. Compared to other competing technologies, low power consumption, small sensor and sample volumes (e.g. blood), fast response times, excellent manufacturing/process repeatability and mass production capability with high production yield are strong advantages of microsensor technology. Micro electro-mechanical biosensors provide an interesting means to analyze the content of a biological sample based on the direct conversion of a biological event to an electronic signal. They

allow highly sensitive detection of micro-organisms or toxins with high selectivity using minimal sample quantity.

Before getting involved in the technical aspects, it is essential to understand the latest development trends in biosensing. In Figure 1-1, the sales data for various MEMS components in the life sciences market are shown. BioMEMS are increasingly being used in DNA chips, lab-on-a-chip, biosensors, drug delivery systems and implants.

Figure 1-1 MEMS components for life sciences global market trend [3]

Demand for sensors in the US will rise 6.1 percent annually to 2014 [4] and it is predicted to expand continuously in the future, mainly driven by medical and diagnostics applications. These provide good justifications for our biosensor study.

Biosensors themselves can be classified into four main groups based on different principles of transduction [5, 6]: 1) Thermal sensors, 2) Chemomechanical sensors, 3) Optical sensors and 4) Electrochemical sensors. Each sensing mechanism has its

particular advantages and disadvantages with respect to the sensitivity, selectivity, resolution, manufacturing cost, robustness to disturbances, etc. Figure 1-2 shows a general overview of the chemical sensors classification.

Figure 1-2 Classification of the chemical/biochemical sensors

1.2 Research Objectives

In many biological applications, high sensitivity mass sensors are required to capture small biological entities. For instance, a highly sensitive resonator with high Q-factor response is required to capture the biomarkers proteins in low concentration solutions. The main objective of this project is to develop a high performance resonator for chemical/biochemical applications. Microresonator structures fabricated on silicon

substrates are mainly investigated in this work to be able to benefit from well-established MEMS technologies for sensor array integration in the future. In this work, two particular resonator types are designed, fabricated and their mass sensitivities characteristics are studied. The first designed resonator is a single-crystal silicon squared resonator with dielectric filled capacitive excitation mechanism. The second resonator is a piezoelectric AlN resonator with checker-patterned electrode architecture. The mass sensitivity of the developed sensor is analytically derived and confirmed with FEM simulations and experimental results. The obtained high mass sensitivity of the developed sensors are much higher than state of the art mass sensors and shows the high potential of the developed piezoelectric transducers for mass sensing applications. Finally, the application of the designed resonator as a biological sensor in liquid environment has been demonstrated experimentally using the specific binding of biotin and streptavidin immobilized on the resonator surface.

1.3 Organization

After the introduction, MEMS-based sensor technologies for chemical and biochemical sensing applications are briefly reviewed and compared in chapter 2. The discussion includes calorimetric, chemomechanical, optical and electrochemical sensors. In this work, two particular resonator types (Lamé-mode resonator and AlN resonator) are designed, fabricated and their mass sensitivities characteristics are studied and compared in both gaseous and liquid environments to be used in biochemical detection application. In chapter 3, the design and modeling of Lamé-mode square disk resonator is discussed. First the work principle of square resonator is generally explained. Subsequently, an

analytical model is derived to estimate the mass sensitivity of the designed sensor. Then, an electric equivalent circuit for the sensor is introduced and the required equations are derived. The fabrication process flow of the resonator is explained followed by the description of the measurement setup. Eventually, the analytical models for the mass sensitivity of the resonator are compared with experimental results and finite element simulations. Chapter 4 introduces a developed piezoelectric AlN checker-mode resonator. Resonator structure design and simulation are described first. Then, the mass sensitivity derivation of checker-mode resonator along with the related simulations and a brief description of limit of detection are discussed. MEMS micromachining processes used to fabricate the resonators followed by the polyelectrolyte multilayer properties and its coating method together with experimental results of sensor characterizations. In chapter 5, the application of the designed Lamé-mode resonator as a biological sensor in liquid environment has been demonstrated experimentally using the specific binding of biotin and streptavidin immobilized on the resonator surface. Finally, chapter 6 concludes the thesis with suggestions for future works.

Chapter 2

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2. Literature Review

Micromachined chemical/biochemical sensors typically consist of a sensitive layer and a transducer, converting changes in the physical or chemical properties of the sensing layer into a signal, generally an electrical signal. Upon exposure to a specific chemical species, the physicochemical properties of the sensitive coating, such as mass, optical properties, resistance, enthalpy and potential, are changed by absorption/adsorption of analyte, chemical reaction, charge transfer, etc [7-9].

In many biological applications, high sensitivity mass sensors are required to capture small biological entities. Advances in micro- and nanofabrication technologies are enabling a wide range of new technologies, including the development of acoustic wave resonators with high potential for very high mass sensitivity [2, 10, 11]. To cover existing technologies for biosensors, four main groups of chemical sensors as shown in previous section i.e. (I) Thermal sensors (calorimetric), (II) Chemomechanical sensors (mass-sensitive sensors), (III) Optical sensors and (IV) Electrochemical sensors are reviewed.

2.1 Thermal Sensors (Calorimetric Sensors).

Thermal or calorimetric sensors work based on the measurement of enthalpy changes of chemical or analyte absorbed in the sensor's surface. Changes in enthalpy lead to changes in temperature which can be converted to an electrical signal. To obtain maximum sensitivity, the sensing part should be insulated as much as possible by, for example,

putting it in an insulated microstructure. The temperature changes generated are then transformed to an electrical signal by a platinum resistive sensor or a thermoelectric sensor [7, 9]. Thermal sensors themselves can also be classified a thermoelectric sensor and catalytic thermal sensor based on the transduction path from heat to the electrical signal.

Thermoelectric sensors were applied for detecting organic volatiles in the gas phase and measuring concentrations of glucose, urea and hydrogen peroxide in the liquid phase [12]. Catalytic thermal sensors are typically used for detecting flammable gases such as methane, butane, hydrogen, carbon monoxide, propane or propylene in gaseous environments [13]. These sensors however generally suffer from a poor selectivity and their application in liquid environment has not been reported yet.

2.2 Electrochemical Sensors

The first electrochemical sensor was invented in 1950s for the purpose of oxygen monitoring [14]. Later, this sensor was used in confined space applications for monitoring toxic and combustibles gases. Later, miniaturizing of electrochemical sensors helped to improve the sensitivity and the selectivity of these types of sensors. Currently, a wide range of electrochemical sensors are being used in many stationary and portable applications for personal safety [7, 9].

An electrochemical sensor is often comprised of two electrodes, a sensing and a reference electrode which are separated by a chemically sensitive layer. Electrochemical reactions or charge transfer at the electrode surface are transduced into an electrical signal, generally a current, potential or conductance. Depending on the electro-analytical

principles, electrochemical sensors are classified into three categories: amperometric, conductometric, and potentiometric sensors. These biosensors are typically used for glucose, fructose, sucrose, urea, cholesterol and ethanol, as well as tissue and whole organisms [15].

2.3 Optical Sensors

The optical biological sensors relate modulation of a transducer's optical characteristics to biological interactions on the sensor surface. In fact, analyte-induced dielectric constant changes of the material in the sensing region modulate the propagation constant of light. Changes in the real part of the dielectric constant, which are related to the reflective surface properties, modulate the speed of light propagation. On the other hand, changes in the imaginary part of the dielectric constant, which are related to absorption losses in the medium, result in the attenuation of the light propagation [16, 17]. The classification of optical chemical/biochemical sensors into a few types is difficult because of the wide variety of sensing schemes. According to Gorton [16], the optical sensors can be classified into three main categories: fiber optical (FO) sensors, surface plasmon resonance sensors (SPR) and integrated sensors using planar/channel waveguides.

Optical fiber based biochemical sensors have several advantages including the light transmission over a long distance without severe loss through the optical fiber, thus enabling remote monitoring of chemical species. Secondly, the sensing principle is immune to electromagnetic interference at the sensing site and can be used in harsh environments. Multiple analyte detection or single analyte monitoring at different locations is possible as optical sensors can guide light of multiple wavelengths at the

same time and in different directions. Considering these advantages, optical biosensors have some drawbacks as they are complicated/expensive sensing system in addition to the signal drift by ambient temperature; light and unstable light source/detector [18-20].

2.4 Chemomechanical Sensors

Chemomechanical sensors measure the mechanical property changes of sensor such as mass, elastic modulus and deflection when a secondary material is deposited on their surface. Cantilever sensors are the most common types among others used in airbag system [21]. Chemomechanical sensors are generally classified into static and dynamic types depending on the mode of sensor operation.

2.4.1 Static-Mode Sensors

Static-mode chemomechanical sensors generally determine the mass concentration by measuring the deflection in a microcantilever or diaphragm from the amount of induced stress. The microcantilever or diaphragm is modified so that one surface is highly active (high affinity to the target analyte) and the other surface is passive. The static mode detection method has been used for detecting various biochemical species in the liquid phase, such as glucose [22] and myoglobin [23], complementary strands of oligonucleotides [24] and prostate-specific antigen (PSA) [25], showing sensor resolutions of 0.2 ng/ml for the prostate-specific antigen (PSA) and 20 µg/ml for myoglobin. Piezoresistive detection of the microstructure is one of the successful applications of static-mode sensors [26]. It requires only silicon piezoresistors which are readily available from most silicon-based microfabrication processes. Because of the

large piezoresistive effect in single crystalline silicon, the piezoresistive detection method is widely applied in silicon-based transducer design [27-29].

2.4.2 Dynamic-Mode Sensors

Dynamic-mode sensors measure the frequency shift due to change of mass in the resonating structure. The output frequency is not sensitive to variations of signal amplitude and can be easily measured by an analyzer [30].

2.4.2.1 Dynamic-mode sensors based on the excitation mechanism

Dynamic mode sensors can be viewed and classified based on their excitation mechanism. Most common mechanisms are piezoelectric, electrostatic, electrothermal and magnetoelastic, respectively.

Piezoelectric driving mechanisms are widely used in acoustic-wave devices [31] but require using either piezoelectric substrates or the deposition of a layer of piezoelectric thin film. Electrostatic excitation is common in high-frequency resonators and micro-fabricated vibratory gyroscopes. However, the required submicrometer air gaps result in unwanted squeezed film damping and might be clogged in dirty environmental in biochemical applications [32-34].

Magnetoelastic sensors are used for detection of food borned pathogens, measure or monitor various physical properties (e.g., pH and pressure) and blood coagulation behavior [35, 36]. Electrothermal excitation, has been shown to be effective and simple

requiring only heating resistors [7]. Chemomechanical sensors (based on their excitation mechanisms) are listed in Table 2-1 together with some of their common applications.

Table 2-1 Comparison of dynamic chemomechanical biosensors based on their excitation mechanisms

	Type	Application	References
Chemomechanical Sensor (Based on Excitation Mechanisms)	Piezoelectric	Common in acoustic-wave devices, Wireless applications, Biosensors	[31], [37], [38], [39]
	Electrostatic (Capacitive)	In high frequency resonator, Comb drive	[34], [33], [40].
	Magnetoelastic	Detection of food borne pathogens, measure or monitor various physical properties (e.g., pH and pressure) and blood coagulation behavior.	[35], [36], [41], [42]
	Electrothermal	Effective and simple in fabrication	[7], [43], [44]

2.4.2.2 Dynamic-mode sensors based on the vibrating mode

Depending on the modes of wave propagation through or on a substrate, dynamic-mode sensors can generally be classified as Bulk Acoustic Wave (BAW) devices [45, 46] and Surface Acoustic Wave (SAW) devices [30, 47]. The most common BAW and SAW devices and their vibration modes are depicted in Figure 2-1.

Figure 2-1 Wave propagation of dynamic mode sensors, Courtesy of A. Janshoff et al [31]

Each sensor has its own advantages and disadvantages. Among the above resonators, the Quartz-Crystal Microbalance (QCM) or TSM¹ is the best example of resonant mass sensor that has been commercialized for film thickness monitoring and other mass sensing applications [30, 48]. Its high frequency stability allows the detection of very small frequency shift providing limits of mass detection in the order of a few nanograms. The QCM, however, is limited in its monolithic integration with electronics as well as relatively low frequency that does not allow these devices for the detection of very small quantities of volatile organic chemicals or warfare agents. Despite the extensive use of QCM technology, some challenges such as the improvement of the mass sensitivity remains unsolved [30, 48]. Recently an electrodeless QCM biosensor with high mass sensitivity has been reported [49]. The result shows that the classical QCM technique still remains a promising technique. Once this the problem of low mass sensitivity is solved,

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¹ Thickness Shear Mode (TSM) resonator are also known as Quartz-Crystal Microbalance (QCM).

the next challenges would be the high cost due to their complex manufacturing and the difficulties in applying them for sensor arrays due to lack of integration capability and appropriate characterization interface [50].

SAW devices have been also attracting the attentions of researchers, as some of these devices (like the Love Wave), have demonstrated higher mass sensitivity in liquid media, than the traditional QCM based sensors. In general, SAW devices operate with higher frequencies, leading to higher sensitivities [51, 52]. Another approach used to get higher mass sensitivities is through film bulk acoustic resonators (FBAR) devices. FBAR could also provide high mass sensitivities and extraordinary limit of detection (in atto or zepto gram ranges) due to the higher resonance frequency and miniaturized structures [53-55]. However, as the frequency of the FBARs is set by the piezoelectric layer thickness, even if they can guarantee high mass sensitivities, they cannot provide for multiple frequencies operations (and therefore sensitivities) on the same chip. Some of the above-mentioned specifications (resonance frequency and mass sensitivity), advantages and disadvantages of these sensors are summarized in Table 2-2.

Table 2-2 Comparison of the most common acoustic wave devices

Device		Frequency [MHz]	Sensitivity [$\mu\text{m}^2/\text{ng}$]	Advantages	Disadvantages
BAW	TSM (QCM)	5-100	1.4	Robust Nature, Availability, High limit of detection.	Limited frequency range, Low Q-factor, Fragility during fabrication.
	SH-APM	5-200	1.5	Design to operate in liquid.	—
SAW	Rayleigh	30-300	13.3	Working in gas.	—
	SH-SAW	30-300	18.0	—	Higher energy loss in aqueous environment.
	Love Wave	30-300	22.1	High mass sensitivity.	—
	FPW	5-20	49.0	High Q-factor, Low energy loss in liquid at a low resonant frequency.	—

2.4.3 Comparison of Different Microsensor Technologies

Comparison of different biological sensors is relatively difficult because each sensor generally has its own specific requirements depending on the application area. However, high sensitivity, simple and inexpensive fabrication process, wide sensing range, and the simplicity of the overall sensing system are common features that every microsensor tries to attain. General advantages and applications of all aforementioned sensor types (thermal, chemomechanical, optical and electrochemical sensors) are listed and compared with regard to the above-mentioned aspects in Table 2-3.

Table 2-3 Different bio sensors based on transduction mechanisms for microsensor

Sensor Types		Resolution	Application	References	Advantages	Disadvantages
Thermal Sensors	Catalytic Thermal Sensors	Moderate	Used for detecting flammable gases such as methane, butane, hydrogen.	[26], [13]	Operational stability for continues monitoring, High thermal noise rejection ratio.	Only detect changes of analyte concentration. Sensitive to thermal conductivity of medium.
	Thermoelectric Sensors	Moderate	Detecting organic volatiles in the gas phase and measuring concentrations of glucose, urea and hydrogen peroxide in the liquid phase.	[43], [44] [56]		
Optical Sensors	Fiber Optic Sensors	High	Remote monitoring of chemical species, Using in harsh environments.	[16, 17], [57]	High sensitivity, High selectivity due to spectroscopic methods, Multi-analyte sensing, Fast response time.	Complicated, expensive sensing system. Photobleaching of sensitive layer. Signal drift by ambient temp., light, and unstable light source, detector.
	Surface Plasmon Resonance (SPR) Sensors	High	Gas sensing and biosensing of an antigen-antibody interaction, Using in harsh environments.	[18], [19]		
	Integrated Optical Sensors	High	-----	[20], [58]		
Electro-chemical Sensors	Amperometric Sensors	Moderate	Glucose sensing using immobilized glucose oxidize (GOD) as a biocatalyst.	[15]	Simple structure, Inexpensive fab.	High background noise floor. Sensitive to the quality of sensing layer.
	Conductometric Sensors	Moderate	-----	[9]		
	Potentiometric Sensors	Moderate	Biochemical sensing applications.	[9], [59]		
Electro-Mechanical Sensors	Static-Mode Sensors	High	Detecting various biochemical species in the liquid phase, such as glucose, imyoglobin.	[26], [23], [24]	High sensitivity, Operation in liquid	Long response time. Low signal-noise ratio.
	Dynamic-Mode Sensors	High	Detection of organic volatiles in the gas phase using polymer coatings, biosensing in liquid to detect, e.g., serum albumin.	[30], [60]	High sensitivity and wide dynamic range, Simple fab., Fast response time	Sensitive to thermal noise. Low resolution in liquid.

2.5 Summary

Among all possible transduction mechanisms for biosensors, dynamic-mode chemomechanical sensors have been chosen for this study. In addition to their high mass-sensitivity, any bio-entities that bind to the sensor surface can be detected (label free sensors). Moreover, the frequency output of the resonant sensor makes it insensitive to variations in signal amplitude and can be easily digitized using simple counters [7, 59]. Briefly, compared to other competing technologies, low power consumption, small sensor, fast response times, excellent manufacturing repeatability and mass production capability with high production yield are strong advantages of dynamic-mode chemomechanical sensors.

Chapter 3

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3. Dielectric Filled Lamé-Mode Square Sensor

In this chapter the design, fabrication and characterization of a dielectric Lamé-mode square resonator are presented. The designed resonator is actually a high performance sensor with dielectric filled capacitive excitation mechanism. The resonators are fabricated using the silicon-on-insulator MEMS process. In order to study the mass sensitivity of the fabricated resonator, a polyelectrolyte multilayer (PEM) is used to coat on the resonator surface. The 34.81MHz resonator has the mass sensitivity of $105.4 \mu\text{m}^2/\text{ng}$ and limit of detection of $1.36 \times 10^{-6} \text{ ng}/\mu\text{m}^2$. Experimental measurements of the mass sensitivities agree with theoretical predictions. The developed sensor shows a high potential to be used in bio detection applications.

3.1 Device Design and Simulation

The microresonator consists of a square silicon plate supported by four beams attached to it from the corners (see Figure 4-1(a)). The square plate is separated from the surrounding electrodes by a 80 nm nitride gap that defines the capacitance of the electro-mechanical transducer. The resonator is laterally driven with electrodes on two opposite sides of the square plate symmetrically. A DC-bias voltage (V_p) is applied to the structure via the anchors, while two AC input voltages are applied to the input electrodes with 180 degrees of phase difference. These voltages result in a time-varying electrostatic force on the plate edges which makes it oscillate in its fundamental frequencies of the Lamé-mode. The

capacitive gap distances of the opposite side change with this frequency. Because of the electrostatic field in the gap, a time-varying current is induced in the output electrode. A Cross-sectional view of the square resonator, showing the nitride gap between resonator and its electrodes is depicted in Figure 4-1(b). The filled nitride gap defines the capacitive gap of the input and output capacitors.

Figure 3-1 (a) Schematic of the device showing the resonator, exciting and sensing electrodes, driving and biasing setup (b) Cross-sectional view of the square resonator, depicting the 80nm solid gap between resonator and its electrodes

The square resonator is simulated using COMSOL Multiphysics and the Lamé-mode is shown in Figure 4-2. A mesh study is performed to achieve a converged frequency independent from the mesh size.

Figure 3-2 FEM simulation of square resonator with side length of 100 μm in Lamé-mode in COMSOL-Multiphysics software (The simulated resonant frequency is 38.74 MHz)

In the Lamé-mode, adjacent edges of the square plate bend in anti-phase while the plate volume is preserved. Nodal points of resonance are located at the four corners of the square and at the center of the plate. The obtained resonance frequency in this modelling is for the air-gap resonator. Due to fact that the resonator boundaries are restricted with solid-gap, the resonance frequency will be different. However, the following conditions can be considered for the nitride between the disk resonator and electrodes: (a) the electrodes are moving in unison with the disk itself, while the nitride gap compresses so slightly; (b) the solid nitride gap is not solidly attached to either the disk or electrode, allowing a very small amount of movement across a tiny air gap (a few Angstroms); or (c) the nitride material in the gap is porous, with air bubbles, and the gap compresses and expands while the disk is vibrating. Unfortunately, the zoom-in SEM's are not clear enough to definitively tell which of the above is true. A simulation with solid nitride gap (condition (a)) is also performed and the frequency of 47.12 MHz is obtained (refer to Figure 3-10 (b)). The resonance frequencies from the above boundary conditions (air and solid gap resonators) are good estimations for the experimental resonance frequency.

If the material of the square plate is homogeneous and isotropic, and if the edge of the square L is much larger than its thickness, the square resonator can be theoretically modelled as a thin plate. In this case, the resonant frequency of Lamé-mode can be calculated as [61]:

$$f_0 = \frac{n}{\sqrt{2}L} \sqrt{\frac{C_{44}}{\rho}} \quad (3-1)$$

where n is the order of the resonance mode, ρ is the material density and C_{44} is the stiffness constant which can be replaced by shear modulus term G . For the single-crystal silicon structure, the material properties are assumed as follows:

$$\rho = 2330 \text{ kg/m}^3; G = 70 \text{ GPa}.$$

Replacing these values in Eq. (3-1), the corresponding resonant frequency of a square resonator with length of $100 \text{ }\mu\text{m}$ will be 38.75 MHz .

This particular microstructure has several lower and higher resonance modes at different frequencies (see simulated mode shapes in Figure 3-3), but operation at Lamé mode (shown in Figure 3-2) is preferred for differential measurement. It should be noted that depending on the structure dimensions, the order of the eigenmodes in Figure 3-3 might be changed.

Figure 3-3 Resonance mode shapes of a disk resonator simulated with FEM software COMSOL; the color coding indicates the displacement distribution.

3.1.1 Equivalent Mechanical Model of the Resonator

Similar to all oscillatory systems, the square disk resonator can be represented by a lumped-element equivalent model composed of a rigid-body mass, spring and damper element (Figure 3-4).

Figure 3-4 Mass-spring-damper system model for micromechanical resonator

This simplification helps in designing the resonator and its adjacent electrodes parameters. Additionally, it is possible to use this lumped model to extract the electrical equivalent model later. The equation of motion of the square resonator is:

$$m_{eq}\ddot{X} + c_{eq}\dot{X} + k_{eq}X = F(t) \quad (3-2)$$

where X is the 2-D displacement vector in plane ($X = \begin{bmatrix} x \\ y \end{bmatrix}$), k_{eq} is the equivalent stiffness at the perimeter of the resonator, m_{eq} is the equivalent mass of the resonator, c_{eq} is the damping element and finally $F(t)$ is the electrostatic driving force. The equivalent mass of the resonator is related to the device geometry and is given by

$$m_{eq} = \rho L^2 t \quad (3-3)$$

where t is the resonator thickness. The equivalent stiffness k_{eq} at the perimeter of the resonator is related to angular resonant frequency ω_0 as:

$$k_{eq} = \omega_0^2 m_{eq} \quad (3-4)$$

where $\omega_0 = 2\pi f_0$ (f_0 is the resonant frequency of Lamé-mode and can be found from Eq. (3-1)). The damping element, c_{eq} is also related to k_{eq} , m_{eq} and the Q -factor of the resonator

$$c_{eq} = \frac{\sqrt{k_{eq} m_{eq}}}{Q} \quad (3-5)$$

Generally the theoretical and accurate prediction of Q -factor is difficult due to the complexity in modelling all the loss mechanisms and is typically determined experimentally. In this work, the Q -factor is estimated using simulation model in Section 3.1.2 and also experimentally measured in Section 3.4.

Given a harmonic driving force of $F(t) = F \cdot \cos(\omega t)$, after solving the Eq. (3-2), an expression for the transfer function of displacement to driving force of resonator in phasor form is

$$\frac{X(j\omega)}{F(j\omega)} = \frac{k^{-1}}{(1 - (\omega/\omega_0)^2) + j(\omega/\omega_0)/Q} \quad (3-6)$$

where ω_0 is the natural resonant frequency in radian. The vibration amplitude of resonator in time domain is then:

$$|X| = \frac{F/M}{4\pi^2 \sqrt{(f^2 - f_0^2)^2 - (f \cdot f_0/Q)^2}} \quad (3-7)$$

where f_0 is the resonant frequency. In Figure 3-5, when the amplitude of vibration is plotted against frequency, near the region of resonant frequency (f_0), the amplitude response is enhanced by the quality factor Q , that is, $|x| = QF/k$ at resonance from Eq. (3-6).

Figure 3-5 Vibration amplitude vs. frequency plot of a typical resonator

As mentioned above the resonator of this work uses capacitive transduction to excite the device. In a two-port configuration, a direct DC-bias voltage V_p is applied to the resonator and an alternating voltage AC signal v_i to its input electrode. Note that the DC-bias voltage V_p serves only to charge the electrode-to-disk capacitance, and as a result consume no power. These voltages together generate an electrostatic driving force $F(t)$ in a x (or y) direction given by [62]

$$F(t) = V_p \left(\frac{\partial C}{\partial x} \right) v_i \quad (3-8)$$

where C is the dynamic electrode-to-resonator overlap capacitance, $\frac{\partial C}{\partial x}$ is the change in capacitance per unit displacement in x (or y) direction. Before finding an expression for $\frac{\partial C}{\partial x}$, it is required to introduce the static electrode-to-resonator capacitance as below:

$$C_0 = 4 \varepsilon_r \varepsilon_0 \frac{Lt}{d_0} \quad (3-9)$$

where d_0 is the gap distance between the resonator and its surrounding electrodes (which is filled with silicon nitride), ε_r is the relative permittivity (which is 7.8 for the case of the silicon nitride) and ε_0 is the vacuum permittivity (8.85×10^{-12} F/m). Consequently, the dynamic electrode-to-resonator overlap capacitance C is defined as:

$$C = 4 \varepsilon_r \varepsilon_0 \frac{Lt}{(d_0 - x)} = C_0 \left(1 - \frac{x}{d_0}\right)^{-1} \quad (3-10)$$

where x is the displacement of the edge of the resonator. As a result, driving force $F(t)$ can be obtained by replacing Eq. (3-10) into Eq. (3-8)

$$F(t) = V_p v_i \frac{C_0}{d_0} \left(1 - \frac{x}{d_0}\right)^{-2} = V_p v_i \frac{C_0}{d_0} (1 + A_1 x + A_2 x^2 + \dots) \quad (3-11)$$

where $A_1 = \frac{2}{d_0}$, $A_2 = \frac{3}{d_0^2}$, ... are the coefficients in the Taylor expansion. If displacements are small, this equation can be simplified by considering the first term of the Taylor expansion and deduce to the below simpler form

$$F(t) \cong V_p v_i \frac{C_0}{d_0} \quad (3-12)$$

3.1.2 Q-factor

Depending on the geometry of the resonator and its working environment, various energy losses contribute in total Q which are intrinsic material damping, air damping (Q_{air}), thermo-elastic dissipation (Q_{TED}), anchor losses (Q_{anchor}) and surface loss mechanisms [63-65]. As a result Q_{total} can be expressed as

$$\frac{1}{Q_{\text{total}}} = \frac{1}{Q_{\text{surface}}} + \frac{1}{Q_{\text{TED}}} + \frac{1}{Q_{\text{anchor}}} + \frac{1}{Q_{\text{air}}} + \frac{1}{Q_{\text{other}}} \quad (3-13)$$

where each term represents the contribution to Q_{total} from the corresponding energy loss mechanism. The mechanism with the lowest Q is the dominant loss mechanism. Q_{surface} is considered in resonators with small surface-to-volume ratios, which is only correct for large bulk-mode resonators. In this section, the Q -factors related to air damping (Q_{viscous}) and thermoelastic damping (Q_{TED}) mechanisms are simulated in order to have an estimation for the sensor design.

3.1.2.1 Viscous Damping

In structural engineering, viscous, velocity-dependent damping is very difficult to visualize for most real structural systems. Only a small number of structures have a finite number of damping elements where real viscous dynamic properties can be measured. In most cases modal damping ratios are used in the computer model to approximate unknown nonlinear energy dissipation within the structure.

Losses due to air damping can be divided to squeeze film damping and slide film damping. As the squeeze film damping is eliminated in our design (air-gaps are filled

with nitride), the slide damping on the top and bottom surfaces of the resonator is the only source of air damping. Consequently, this model focuses on slide film damping as the main energy damping sources. A theoretical model [66] and a FEM model are presented to calculate the slide film-damping in the square resonator.

i) An analytical model of slide film damping

Micromechanical devices fabricated by surface micromachining technology feature thin movable plates (about 2 μm thick) suspended with a small gap over a substrate by flexures. This basic structure facilitates the lateral movement of the plates for such applications as resonators, actuators, accelerometers, etc.

As the dimensions of the moving plates are usually much larger than their thickness and the gap distance, the viscous damping by the ambient air plays a major role in energy dissipation of the dynamic system; the air film behaves as a slide-film damper to the moving structure. To investigate the basic features of slide-film damping, a mechanical model is considered: an infinite plate, immersed in an incompressible viscous fluid, moving in a lateral direction at a constant distance from the substrate as shown in Figure 3-6 (a) [67]. The simplified model is shown in Figure 3-6 (b).

Figure 3-6 –Slide-film damping (a) schematic structure; (b) a simplified model

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The general differential equation for the steady flow of an incompressible fluid is the well-known Navier-Stokes equation [68]

$$\rho \left[\frac{\partial \vec{v}}{\partial t} + (\vec{v} \cdot \nabla) \vec{v} \right] = \vec{F} - \nabla p + \mu \nabla^2 \vec{v} \quad (3-14)$$

where ρ is the density of the fluid, $\vec{F}$ the force applied, p the pressure in the fluid, μ the coefficient of viscosity and $\vec{v}$ the velocity of the fluid

$$\vec{v} = u\vec{i} + v\vec{j} + w\vec{k}$$

The notations ∇ and ∇^2 are gradient and Laplace operators, respectively,

$$\nabla = \vec{i} \frac{\partial}{\partial x} + \vec{j} \frac{\partial}{\partial y} + \vec{k} \frac{\partial}{\partial z} \text{ and } \nabla^2 = \frac{\partial^2}{\partial x^2} + \frac{\partial^2}{\partial y^2} + \frac{\partial^2}{\partial z^2}$$

For example, the Navier-Stokes equation for the x-direction is

$$\rho \left[\frac{\partial u}{\partial t} + \left(u \frac{\partial}{\partial x} + v \frac{\partial}{\partial y} + w \frac{\partial}{\partial z} \right) u \right] = F_x - \frac{\partial p}{\partial x} + \left(\frac{\partial^2}{\partial x^2} + \frac{\partial^2}{\partial y^2} + \frac{\partial^2}{\partial z^2} \right) u$$

Suppose that the plate is in the x - y plane of the coordinate system and infinite in dimensions. If the movement of the plate is in the x -direction, and there is no external force or pressure gradient in the fluid, then $\frac{\partial^2 u}{\partial z^2} \gg \frac{\partial^2 u}{\partial x^2}, \frac{\partial^2 u}{\partial y^2}$. Thus, Eq. (3-14) reduces to

$$\frac{\partial u}{\partial t} + u \frac{\partial u}{\partial x} = \frac{\mu}{\rho} \frac{\partial^2 u}{\partial z^2} \quad (3-15)$$

For an infinite plate (i.e., the plate is much larger than the gap distance between the plate and the substrate and its oscillation amplitude), the second term on the left side of Eq. (3-15) may be neglected, resulting in

$$\frac{\partial u}{\partial t} = \frac{\mu}{\rho} \frac{\partial^2 u}{\partial z^2} \quad (3-16)$$

The non-slippage boundary conditions *for* the equation are: u equals zero at the substrate surface and u equals the velocity of the moving plate on the surface of the plate. Now let us discuss more on the approximation conditions for Eq. (3-16). Suppose that the motion of plate with reference to its balanced position is a simple harmonic oscillation

$$x(t) = a_0 \sin \omega t$$

where a_0 is the amplitude of the simple harmonic oscillation, i.e.,

$$u(t) = a_0 \omega \cos \omega t = u_0 \cos \omega t$$

where $u_0 = a_0 \omega$. Thus, the first term on the left of Eq. (3-15) is

$$\frac{\partial u}{\partial t} = -u_0 \omega \sin \omega t \quad (3-17)$$

If the typical dimension of the plate is l , the second term on the left of Eq.(3-15) is

$$u \frac{\partial u}{\partial t} \approx \frac{u_0^2}{l} = \frac{a_0^2 \omega^2}{l} \quad (3-18)$$

and the term on the right is

$$\frac{\mu}{\rho} \frac{\partial^2 u}{\partial z^2} \approx \frac{\mu}{\rho} \frac{a_0^2 \omega^2}{l} \quad (3-19)$$

where d is the gap distance between the substrate and the moving plate. Therefore, the approximation conditions for Eq. (3-16) are

- a) $\left| \frac{\partial u}{\partial t} \right| \gg \left| u \frac{\partial u}{\partial x} \right|$. This requires small amplitude. i.e., $a_0 \ll l$,
- b) $\frac{\mu}{\rho} \frac{\partial^2 u}{\partial z^2} \gg u \left(\frac{\partial u}{\partial x} \right)$.

From Eqs. (3-18) and (3-19), this condition becomes $l \gg 2 (\rho\omega/2\mu)d^2a_0$. By defining a characteristic “effective decay distance” $\delta = \sqrt{2\mu/\rho\omega}$, then

$$l \gg 2 \frac{d^2}{\delta^2} a_0 \quad (3-20)$$

The curve in Figure 3-7 shows the dependence of δ on the frequency in air at 1 atm at 20°C. In the condition of $\frac{\mu}{\rho} \frac{\partial^2 u}{\partial z^2} \gg (\frac{\partial u}{\partial t})$, i.e., $d \ll \delta$, Eq. (3-16) is further simplified as

$$\frac{\partial^2 u}{\partial z^2} = 0 \quad (3-21)$$

Figure 3-7 The effective decay distance, δ , as a function of frequency [66]

Suppose that a large plate oscillates laterally over a substrate as shown in Figure 3-8. If the oscillating frequency is low so that $\delta \gg d$, the flow pattern of the air around the plate is called a Couette-flow [66]. The damping force on the plate is considered to be caused by the viscous fluid by the Couette-flow model with the non-slippage boundary conditions

$$u(0) = u_0 \cos \omega t, u(d) = 0 \quad (3-22)$$

Figure 3-8 Laterally oscillating plate over a substrate

According to Eqs. (3-21) and (3-22), the velocity distribution of the fluid is

$$u(z) = u(0)\left(1 - \frac{z}{d}\right) \quad (3-23)$$

where $u(0)$ is the velocity of the moving plate. The resistive shearing force applied to the plate is

$$F = -\mu \frac{u(0)}{d} A \quad (3-24)$$

where A is the area of the plate. According to the Couette-flow model, the velocity gradient on the open side (top) of the plate is zero. Therefore, there is no damping force on the top of the plate and the Q factor of the lateral vibration system is determined only by the damping force described in Eq. (3-23). (As a matter of fact, if d on the top is large, the condition for Couette-flow, $\delta \gg d$, is no longer valid, but it is just assumed here that the damping force on the top of the plate is negligible.)

The energy dissipated by the damping force in one cycle is

$$\Delta E_{viscous} = \int_0^{2\pi} A\mu \frac{u(0)}{d} u(0) dt \quad (3-25)$$

As $u(0) = u_0 \cos \omega t$, then:

$$\Delta E_{viscous} = \int_0^{2\pi} A\mu \frac{u_0^2 \cos^2 \omega t}{d} \frac{1}{\omega} d\omega t = \frac{\pi}{\omega} u_0^2 \frac{u}{d} A \quad (3-26)$$

According to the definition of Q factor ($Q = 2\pi \frac{E}{\Delta E} = \frac{m\omega_r}{c}$):

$$Q_{viscous} = \frac{\pi m u_0^2}{\Delta E_{cd}} = \frac{m\omega d}{\mu A} \quad (3-27)$$

If the mass density of the plate is ρ_p and the thickness of the plate is H , Eq. (3-27) can be written as

$$Q_{viscous} = \frac{\rho_p H \omega d}{\mu} \quad (3-28)$$

Note that $Q_{viscous}$ is not dependent on the area of the plate, A . The parameters of the Lamé-mode square resonator plate and surrounding air in room temperature are listed as below:

$$\rho = 2330 \text{ kg/m}^3; H=2 \text{ }\mu\text{m}; \omega = 2.39 \times 10^5 \text{ rad/s}; d = 2 \text{ }\mu\text{m}; \mu = 18 \times 10^{-6} \text{ Pa.s}$$

Then, the $Q_{viscous}$ of the resonator is obtained to be 1.59×10^5 .

ii) FEM simulation of slide film damping

A schematic cross section of resonator, nitride gap and electrode together with damping mechanisms and boundary conditions are shown in Figure 3-9.

Figure 3-9 Schematic of the cross section of the resonator, nitride gap, surrounding electrodes, damping mechanisms and boundary conditions

To evaluate the Q -factor, an eigen-value analysis is firstly carried out to obtain the Lamé-mode resonant frequency. The meshed model and the resonator in the Lamé-mode are shown in Figure 3-10.

Figure 3-10 FEM simulation of resonator in COMSOL Multiphysics (a) The meshed model of the resonator and its electrodes; (b) The mode shape simulation of the solid-gap resonator in the Lamé-mode ($f_0=49.12$ MHz)

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Once the resonant frequency is known, the slide-film damping is added to the top and bottom surfaces of the resonator using the boundary condition definition in the software. For this purpose, a 2 μm thick gas film to the top and bottom of the resonator is assumed to contain air at atmospheric pressure. The parameters used for describing the film behaviour are listed in Table 3-1:

Table 3-1 Parameters used for the air layer to simulate the slide film damping

Parameters	Symbol	Value
Film thickness	d	2 μm
Ambient pressure	p_A	1 atm
Mean free path at 1 atm	λ_0	70 nm
Dynamic viscosity	μ	$18 \times 10^{-6} \text{ Pa.s}$

Finally, a frequency response analysis for frequencies in the close vicinity of the resonant frequency is performed. The Q -factor can be estimated using the below equation:

$$Q = \frac{2\pi W_0}{\Delta W} \quad (3-29)$$

where W_0 is the stored vibrational energy, ΔW is the energy lost per cycle. The vibrational energy of the resonator is proportional to the peak velocity of the proof mass. This point helps to estimate the Q -factor directly from velocity of one point at the edge of resonator. After doing a frequency sweep with high resolution close to the resonant frequency, the squared velocity of point A (shown in Figure 3-10 (a)) is plotted in Figure 3-11. Considering steady-state driven oscillations, the Q -factor is commonly defined as the resonance frequency divided by the bandwidth ($Q = \frac{f_0}{\Delta f}$), where the bandwidth Δf is the half-power width of the spectrum.

Figure 3-11 The resonance peak of point A velocity (The Q value is obtained with measuring the resonant frequency divided by the band width at half power)

After calculation on the graph, the estimated Q is obtained to be Q_{viscous} (1.93×10^5). The obtained value from the simulation agrees with the theoretical Q_{viscous} (1.59×10^5). It should be considered that the Q -factor obtained from this simulation is only an upper bound for the considered design and all other damping mechanisms work to further decrease the Q -factor. Therefore, if the slide film damping can be assumed to be the dominating dissipative process in the system, optimizing the Q value obtained from this simplified model will be a good starting point for improving the design.

3.1.2.2 Thermoelastic Damping (TED)

Thermoelastic damping is an important factor that the resonator designer needs to consider [63, 69, 70]. It is a result of a phenomenon called thermoelastic friction, which takes place when materials are subjected to cyclic stress. The stress results in deformation, and the required energy is mostly stored as internal potential energy. However, materials heat under compressive stress and cool under tensile stress. Thus, due

to the heat flow from warmer to cooler regions, energy is lost as nonrecoverable thermal energy. The amount of thermoelastic friction and damping depends on the rate of this energy loss. The magnitude of the energy loss depends on the vibrational frequency and on the structure thermal relaxation time constant, which is the effective time the material requires to relax after an applied constant stress or strain. Therefore, the effect of thermoelastic dissipation, and consequently the damping, is most pronounced when the vibration frequency is close to the thermal relaxation frequency.

For simple structures, researchers have developed analytical expressions to estimate thermoelastic damping. According to Duwel et al [71], one can calculate the Q_{TED} value for a resonator with a single thermal mode by:

$$\frac{1}{Q} = \left(\frac{E \alpha^2 T_0}{\rho C_p} \right) \left(\frac{\omega \tau}{1 + (\omega \tau)^2} \right) \quad (3-30)$$

where E is the Young's modulus, α is the thermal expansion coefficient, T_0 is the resonator temperature at rest, ρ is the density, C_p is the heat capacity of the material, ω is the vibration angular frequency, and τ is the thermal relaxation time of the system. Thus it is easy to see that in order to have better Q value, the system needs to be designed so that ω is as far from $1/\tau$ as possible. The problem is that Equations (3-30) is valid only for very simple structures. Therefore more advanced methods, such as FEA, are required to model more complicated model like our design (Lamé-mode resonator). COMSOL Multiphysics is used for this purpose. To model thermoelastic damping, both thermal and structural problems should be considered simultaneously with a 2-way coupling between them. The strain rate heats or cools the material locally which produces thermal strains. Figure 3-12 shows the geometry and dimensions of the resonator. The side length and thickness of the modelled resonator are 100 μm and 2 μm , respectively.

Figure 3-12 The geometry and the boundary conditions of the modeled resonator in COMSOL Multiphysics for simulating thermoelastic damping Q_{TED}

One factor that affects the simulated Q value is the boundary condition for the thermal equation. In this simulation, thermal insulation applied on all boundaries, but it can be assumed that there is a flux of heat at least from the anchors ends of the resonator. If constant temperature ($T = 0$) assign to the anchors ends, the Q value improves considerably. The physically correct result should be somewhere between these two estimates. Table 3-2 lists the physical properties of the resonator material and the ambient used in this simulation:

Table 3-2 Material properties of silicon-single crystal used in TED simulation

Property	Symbol	Value
Young's modulus	E	169 GPa
Density	ρ	2330 kg/m ³
Poisson's ratio	ν	0.3
Thermal expansion coefficient	α	$2.6 \times 10^{-6} \text{ K}^{-1}$
Specific heat	C_p	700 J/(kg·K)
Thermal conductivity [300K]	k	148 W/(m·K)
Ambient and initial beam temperature	T_{init}	300 K

To obtain Q_{TED} , it is required to find the natural frequency of the modeled resonator first. An eigenfrequency analysis is performed to find the eigenvalues for this system. Figure 3-13 shows the eigenmodes and temperature distribution corresponding to the obtained eigenvalue. As expected from the theory, the temperature is higher near the compressive strain and lower near the tensile strain. The resonance frequency and Q are obtained to be 37.34 MHz and 5.48×10^5 , respectively. The obtained result from simulation is consistent with estimation from Zener theory using Eq. (3-27). According to these results, Q_{TED} does not contribute significantly to overall energy damping for this resonator.

Figure 3-13 The desired eigenmode and temperature distribution of the 2D model of Lamé- mode resonator

Note that the displayed temperature range (-9.49°K to 9.49°K) should not be taken literally because the software normalizes the solution from the eigenfrequency solver. The obtained results of air-damping and thermoelastic damping simulations and the calculated Q_{Total} based on Eq. (3-13) are listed in Table 3-3.

Table 3-3 The total Q-factor of the resonator estimated with simulation

Q_{Viscous}	Q_{TED}	Q_{Total}
1.93×10^5	5.48×10^5	1.88×10^5

3.1.3 Equivalent Electrical Model of the Resonator

The mentioned mechanical model in Sec. 3.1.1 can be transformed into an equivalent electrical circuit. Figure 3-14 shows the equivalent RLC circuit modeling of the square disk resonator of Figure 3-1(a) in a two-port network configuration.

Figure 3-14 Equivalent electrical model for a square resonator using its lumped mechanical model (equivalent values of mass, stiffness and damping)

In this circuit, the motional elements r_x , l_x and c_x model the resonant behavior of the resonator and are directly related to the mass, spring and damper values, respectively:

$$\begin{aligned} r_x &= c_{eq} \\ l_x &= m_{eq} \\ c_x &= \frac{1}{k_{eq}} \end{aligned} \tag{3-31}$$

The transformers in Figure 3-14 model the electromechanical transduction at each port, while the capacitor C_0 models the static capacitance between the n th port (input and output ports; $n=1,2$) and the resonator body, consisting largely of the static capacitance between the electrode and disk but also including any additional parasitic capacitance between the ports and AC ground. The transformer at each port is characterized by an electromechanical coupling coefficient given by

$$\eta_n = V_p \frac{\partial C_n}{\partial x} \quad (3-32)$$

where $\frac{\partial C_n}{\partial x}$ can be obtained from Eq. (3-10). Electrical circuit of Figure 3-14 can be simplified as depicted in Figure 3-15:

Figure 3-15 Simplified electrical model of the two-port resonator in Figure 3-14 driven with a source of impedance R_s , and driving a load of impedance R_L .

The two-port network is driven with a source of impedance R_s , and driving a load of impedance R_L . The element values for this circuit are obtained from the mechanical lumped element values of Eq.(3-31) by reflecting them through the transformers

$$\begin{aligned} R_x &= \frac{r_x}{\eta^2} = \frac{c_{eq}}{\eta^2} \\ L_x &= \frac{l_x}{\eta^2} = \frac{m_{eq}}{\eta^2} \\ C_x &= c_x \cdot \eta^2 = \frac{\eta^2}{k_{eq}} \end{aligned} \quad (3-33)$$

where $\eta = \eta_1 = \eta_2$ because of the symmetry in the Lamé-mode resonator geometry.

Using Eqs. (3-5), (3-32) and (3-33), the motional resistance, R_x corresponds to:

$$R_x = \frac{k_r}{\omega_0 Q} \cdot \frac{1}{V_p^2} \cdot \frac{d_0^4}{\epsilon_r^2 \epsilon_0^2 L^2} \quad (3-34)$$

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When the electrode-to-resonator gap is filled with a dielectric material like nitride, a stiffness quotient should be applied to above equation. This quotient empirically extracted for the nitride in ref. [72]. As a result the final form of motional resistance of nitride filled transducer is:

$$R_{xN} = R_x \cdot \frac{1}{\gamma} \quad (3-35)$$

where $\gamma = 7.6 \times 10^{-4}$ is a stiffness quotient between the nitride gap and surrounding electrode plates. The derived analytical equations for the equivalent electrical model are numerically solved in MATLAB software. Electrical characteristics of the device are calculated for a set of given set of parameters and listed in Table 3-4.

Table 3-4 Input parameters and electrical resonator characteristic at $V_p = 40V$.

		Symbol	Value	Unit
Input Parameters	Elastic modulus of Silicon	E_{Si}	169	[GPa]
	Elastic modulus of Nitride	E_N	270	[GPa]
	Density of Si	P_{Si}	2330	[kg/m ³]
	Density of Nitride	ρ_{SiN}	3184	[kg/m ³]
	Resonator Length	L	100	[μm]
	Anchor width	W_a	4	[μm]
	Electrode gap	d_0	40	[nm]
	Wafer Thickness	t	2.5	[μm]
Electrical Characteristics	Equivalent Mass	m_{eq}	58.25	[pg]
	Equivalent Stiffness	k_{eq}	3.45	[MN/m]
	Q-factor	Q	193000	-
	Motional capacitance	C_x	0.86	[pF]
	Motional resistance	R_x	32.17	[Ω]
	Motional impedance	L_x	0.19	[μH]

In comparison with the previous air-gap resonators [73, 74], the demonstrated resonator provides a much lower motional resistance, R_x . This improvement in motional resistance initiates from both higher dielectric constant (7.8) provided by a nitride electrode-to-resonator gap and the ability to achieve smaller solid gap (up to 20 nm nitride gap in [72] rather than 100 nm of air-gap in [74]). Filling the gap also provides some other benefits since it better stabilizes the resonator structure against shock and eliminates the possibility of particles or liquid getting into the electrode-to-resonator air gap, which poses a potential reliability issue.

3.2 Fabrication Process Flow

The fabrication started with a p-type silicon on insulator (SOI) wafer. The thickness of the device layer in the SOI wafer was 2 μm . SOI wafer technology refers the use of a layered silicon-insulator-silicon substrate in place of routine silicon substrates in fabrication process. They can reduce parasitic device capacitance and thereby improve performance and lead to a precise fabrication process. Another advantage of using SOI wafers is related to their base insulator layer material which can be defined as the etching stop layer for the trench [75]. The specifications of the SOI wafer used in the fabrication process are listed in Table 3-5.

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Table 3-5 The 8" SOI wafer specifications used in fabrication process

Layer	Description
Device Layer:	Orientation: <100>+/-0.5 deg Doping: P/B Resistivity: 0.008-0.02 ohm-cm Thickness: 2.0 +/- 0.5 μm Particles:<10@0.2 μm Flats: Notch Finish: Polished
Buried oxide:	Thermal Oxide Oxide: 2.0 μm +/- 5%
Handle Wafer:	Orientation: <100> Doping: P/B Resistivity: 1-20 ohm-cm Thickness: 700 +/- 25 μm Flatness: <5 μm Bow/Warp: <50 μm Finish: Polished

Then, 300 nm of LPCVD nitride and 150 nm of LPCVD oxide were deposited on a SOI wafer as hard masks for silicon etcher with P5000 tool. The fabrication followed with patterning the resonator and its anchors on the SOI wafer and deposition 20-80 nm thick LPCVD nitride. After that, a layer of 2 μm LPCVD polysilicon was deposited to have the required layer for electrodes. The deposited polysilicon was implanted (Boron, doses of $2 \times 10^{15} \text{ cm}^{-2}$ energy of 180 keV, tilt of 7 degrees) and annealed (N_2 , 1 hour, 950°C). The polysilicon layer was then patterned and etched to define the resonator surrounding electrodes. Then interconnections were formed by deposition, patterning and etching 50 nm and 750 nm of tantalum (for adhesion purpose) and aluminum, respectively. To release the backside of resonator, a DRIE etch was used. The fabrication process flow

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was schematically explained in Table 3-6. The detailed of fabrication process with the applied equipments and recipes used are attached in Appendix A.

Table 3-6 Fabrication process flow for square Lamé-mode resonator

Cross Sectional View	Top View
Step 1- Deposition 3KA LPCVD nitride and 1.5KA LPCVD oxide as hard masks for P5000 and KOH mask for backside etch.	
Step 2 – Etching single crystal silicon and patterning the device layer of SOI wafer using P5000; Mask # 1.	
Step 3- Deposition a layer of (20-80 nm) LPCVD nitride.	

Step 4- Deposition of 2.5 μm LPCVD Polysilicon and Implantation the front side with Boron.

Step 5- Pattern electrodes and Etch the Polysilicon (Stop on the nitride in front side), Mask # 2.

Step 6- Etch 20-80nm thick nitride from the front side (stop on silicon). Confirm with EDX.

Step 7- Deposition of “500 $\text{\AA}$ of Ta+7500 $\text{\AA}$ of Al” on the surface, Patterning the connections pads, Mask # 3.

Step 8- Grinding the wafer to 400μm; Opening the reaction chamber from the backside with DRIE using Mask # 4.

The above-mentioned fabrication process flow of Lamé-mode resonator is summarized in

Figure 3-16.

Figure 3-16 Summarized fabrication process flow of Lamé-mode resonator

To etch the 2.5 μm device layer in the first step of fabrication (Table 3-6), a DRIE (STS-CMOS) was initially used. A general DRIE process cycle contains two steps: etching and passivation. During the passivation step, a polymer layer was coated on the sidewall, which should be thick enough to protect the silicon sidewall from chemical erosion in the following etching step. This step by step passivation and etching process resulted in a etch profile similar to Figure 3-17 (a). As shown, a number of scallops formed because of the multiplexed etch-passivate nature of the DRIE process. These scallops may cause short circuit in the capacitive gap when a thin layer of nitride is deposited. To solve the

problem, P5000 Etcher tool was used. P5000 etcher is a cluster tool, consisting of four, independently-controlled etch chamber modules which surround a central load lock. Chambers are Magnetically-Enhanced Reactive Ion Etch (MERIE) systems, each equipped with optical endpoint detection to allow for more customized etching. As shown in Figure 3-17 (b), the etch profile has extremely improved comparing the DRIE (STS-CMOS) etch profile.

Figure 3-17 Comparing the etch profile using different etch tools (a) DRIE (STS-CMOS) forms scallops (b) Improved etch profile using P5000 etcher tool.

The microstructures were released by DRIE (Deep Reactive Ion Etcher) etching from the back of the wafer. An SEM image of fabricated square resonators is shown in Figure 3-18.

Figure 3-18 (a) SEM image of the fabricated biosensor (top-view); (b) Device cross section showing the released resonator using DRIE

Figure 3-19 presents the SEM image of a fabricated micromechanical Lamé-mode resonator, together with a cross section image of the resonator (Figure 3-19 (b)), nitride gap and its surrounding polysilicon electrode as obtained from transmission electron microscope (TEM).

Figure 3-19 (a) SEM image of the fabricated mass sensor; (b) TEM image of the cross-section of silicon resonator, nitride gap and its surrounding polysilicon electrode

Filling the capacitive gap with silicon nitride helps to achieve smaller gap distance in comparison with air gap counterparts [74]. Filling the gap also provides some other benefits since it better stabilizes the resonator structure against shock and eliminates the possibility of particles or liquid getting into the electrode-to-resonator air gap, which poses a potential reliability issue.

3.3 Derivation of Mass Sensitivity for Lamé-Mode Resonator

The capacitive square resonator presented here actually worked as a mass sensor. The working principle of this sensor is that the resonant frequency of the resonator decreases in response to a mass loading on its surface. Mass sensitivity is defined as the shift in the sensor resonance frequency due to the changes in the mass. A higher mass sensitivity helps to measure smaller masses. The mass sensitivity, S_m of the bulk-wave resonator mass sensor is defined as [76]:

$$S_m = - \lim_{\Delta m \rightarrow 0} \frac{1}{f_0} \frac{\Delta f}{\Delta m} \quad (3-36)$$

where Δm is the external added mass on the surface per unit area of the sensor, due to absorption or thin film deposition and $\Delta f = f - f_0$ is the frequency shift in response to the mass loading. The negative sign in Eq. (3-36) is to be cancelled out with frequency change (Δf) since the resonance frequency decreases after adding the mass on the resonator surface. Equation (3-1), can be modified based on the mass per unit area (m) of the resonator as: $m = \rho t$;

$$f_0 = \frac{n}{\sqrt{2}L} \sqrt{\frac{C_{44}t}{m}} \quad (3-37)$$

where t is the resonator thickness. Taking the derivative of Eq. (3-37) with respect to m , and using this definition again, the following equation is obtained:

$$\frac{df}{dm} = -\frac{1}{2m} \cdot \frac{n}{\sqrt{2}L} \sqrt{\frac{C_{44}t}{m}} = -\frac{f_0}{2m} \quad (3-38)$$

As a result, Eq. (3-36) can be rewritten as below:

$$S_m = \frac{1}{2m} = \frac{1}{2\rho t} \quad (3-39)$$

The definition of the mass sensitivity is not dependent to the lateral dimension, the amount of deposited mass and the material properties of the deposited layer and only depends on the density of the resonator structure material (ρ) and resonator's thickness (t). As a result, a sensor with a thinner structure can have a higher mass sensitivity.

The mass sensitivity value (S_m) for the fabricated devices is analytically evaluated and found to be $107.2 \mu\text{m}^2/\text{ng}$ for a 38.75 MHz resonator ($L = 100 \mu\text{m}$, $t = 2 \mu\text{m}$, $\rho = 2330 \text{ Kg/m}^3$). This value is calculated considering that the sensitive layer is uniformly spread over the entire top surface of the resonator. A 3D Finite Element Method (FEM) analysis has been performed with the aid of COMSOL Multiphysics to validate the obtained analytical values of mass sensitivity (S_m). A thin square-shaped mass is added to the surface of the resonator. Then, the device sensitivity is evaluated by gradually varying the density of the added mass and computing the corresponding linear shift in the resonance frequency. The simulated value of the mass sensitivity is obtained to be $107.2 \mu\text{m}^2/\text{ng}$, which shows a good agreement between analytical and simulated values.

The mass sensitivity of the sensor will be restricted with another characterization identified as limit of detection (LOD). LOD describes the smallest amount of mass that a sensor can detect considering the existing noises (environment and measurement noises). A lower value of LOD means that smaller mass can be detected. The LOD of acoustic mass sensor is given by [43]

$$LOD = \frac{N}{S_m \cdot f_0} \quad (3-40)$$

where N is the noise level of the sensor and is equal to 3 times of the standard deviation of measured resonance frequency of a bare resonator ($N = 3\sigma$) and S_m is the mass sensitivity defined in Eq. (3-39). LOD discussed above is given in terms of detectable mass per unit area. The above values of mass sensitivity and LOD are measured for the fabricated resonators in Section 3.5.

3.4 Resonator Characterization for Mass Sensing Application

This section presents the fundamental characterization of the fabricated square resonators. In order to characterize the fabricated resonators, its surface area should be coated with a controlled mass. In this thesis, Polyelectrolyte Multilayer (PEM) polymer is used for this purpose [77, 78]. After coating the specified amount of mass, a measurement setup is required to measure the new frequency after each layer coating. The required steps for the sensor characterization are explained in the following subsections.

3.4.1 Polyelectrolyte Multilayers Coating

PEM is a polymer which forms by the sequential deposition of two oppositely charged polyelectrolytes (poly sodium 4-styrenesulfonate (PSS) and poly allylamine hydrochloride (PAH)). They have the ability of self-assembling on the surface in a controlled layer-by-layer fashion. PAH is a positive charge polymer and PSS has the negative charge. All PAH, PSS and Sodium chloride (NaCl) are purchased from Sigma–Aldrich Chemie GmbH, Germany.

As PEM can only be coated on the charged surfaces, the silicon resonator surface has to be cured beforehand. In this work, a 2% solution of 3-aminopropyltriethoxysilane (APTES) is used for 2 hours to charge the silicon resonator surface. APTES is a highly effective silane coupling agent that is used on silicon and some other substrates to enhance the adhesion [79]. As the APTES make a positive charged layer on silicon surface, the coating process starts with the negatively charged polymer (PSS) and followed by the positive charged polymer (PAH). Polymer films are built by alternately dipping the chip in aqueous solutions of PAH (1 mg/mL) and PSS (1 mg/mL) for 15 minutes each. After each polymer adsorption, the chip is rinsed two times in deionized (DI) water with the assist of shaker table for 5 minutes. PEM is formed by successive dipping in aqueous PSS and PAH solutions. The required steps for polymer coating on silicon surface are summarized in Figure 3-20.

Figure 3-20 Coating steps of PEM: (1) Cure the resonator surface with APTES (2) Dipping in PSS solution and rinsing in DI water; (3) Dipping in PAH solution and rinsing in DI water.

PAH and PSS layers together referred as one bilayer. Thickness of each polymer bilayer is a function of sodium chloride concentration used in polymer solution preparation. In this work, 50 mM of sodium chloride is used in 50 mL of PEMs solution. Having this concentration, the theoretical thickness of one bilayer of PAH/PSS is ~ 1.7 nm [80]. In order to verify the real thickness of coated polymer, samples are prepared by Focused Ion Beam (FIB), and the thicknesses are measured from TEM images. Figure 3-21 shows a TEM image of the resonator cross-section and the coated polymer thickness.

Figure 3-21 TEM image of the silicon resonator cross-section after coating of 12 PEM bilayers

After coating of 12 bilayers of PEM, the measured thickness of the PEM is 19.5 nm which is in a good agreement with the 20.4 nm of theoretical thickness of 12 bilayers. Having the thickness of each polymer bilayer and the density of 1340 kg/m^3 of the PEM [80], the amount of coated mass per unit area, Δm on resonators can be estimated.

3.4.2 Breakdown Voltage:

The maximum applicable DC-bias in our device is not governed by the pull-in voltage of the air-gap resonators, but rather by the breakdown voltage of the nitride-gap. The dielectric breakdown electric field for nitride is 6-10 MV/cm [72]. So the theoretical breakdown voltages for 20 nm and 80 nm thick nitride gap devices are 12 V and 48 V respectively. However, the breakdown voltage is a function of nitride filler quality. The measured current-voltage graphs of some random devices with 20 nm and 80 nm nitride gaps on the wafer edge are measured and depicted in Figure 3-22 and Figure 3-23, respectively:

Figure 3-22 The breakdown voltage of some random devices with 20 nm nitride gap

Figure 3-23 The breakdown voltage of the four devices with 80 nm nitride gap

According to the obtained I-V graphs, the breakdown voltages of 20 nm and 80 nm thick nitride devices are about 9 V and 40 V, respectively. As expected, the breakdown voltage is a function of nitride filler quality and for instance varied from 8.5 V to 10 V for the 20

nm device depending on the quality of nitride sidewall deposition. It should be noted that 9V is not that much smaller than the 10 V pull-in voltage typical for an 20 nm air-gap disk [73]. Further, if a 20 nm air-gap could be achieved (i.e., could be cleared in a release step), then the pull-in voltage for the associated resonator would likely be lower than the 9 V breakdown of a 20 nm nitride gap. In other words, a solid nitride gap likely allows a higher DC-bias voltage than an equivalent air gap.

3.4.3 Measurement Setup

In this thesis, Agilent 4395A network analyzer is used to test the fabricated sensors. This network analyzer is used to apply an AC voltage to the sensor and measure the output signal. Figure 3-24 shows the fully differential drive-and-sense measurement setup implemented in this work. The electrical elements are placed on a printed circuit board (PCB). The designed PCB layout in ALTIUM DESIGNER software is attached in Appendix B.

Figure 3-24 Differential drive-and-sense measurement setup

The AC drive signal is split into positive and negative signals through a single-to-differential conversion block. The polarization DC voltage, V_P is combined with AC voltage and applied to the resonator. The resonator die is wire bonded to the PCB. The SOI substrate is grounded to reduce the parasitics caused by feed through capacitance. The resonator's output current is sensed using a differential transresistance amplifier setup. Finally, the magnitude and phase of the transmission coefficient, S_{21} is measured with the network analyzer. The setup is shown in Figure 3-1 (a) is simpler alternative to a fully differential sensing method.

3.5 Experimental Results and Discussion

The initial measurements are performed for bare resonators, to get the resonance frequency and the Q-factor. S_{21} parameter is measured for resonator with length of

100 μm and gap distance of 80 nm and the transmission and phase are depicted in Figure 3-25. The resonance frequency of this resonator is measured at 34.81 MHz with a Q value of 1800 in an atmospheric pressure when the resonator is biased with a DC voltage of 20 V and an AC drive voltage of 0.63 V_{pp}.

Figure 3-25 Measured transmission and phase curves of 34.81 MHz Lamé-mode square at atmospheric pressure and bias voltage of 20V DC, 0.63V_{pp} AC and measured Q of 1800

The difference between the theoretical frequency (38.75 MHz) and the measured frequency (34.81 MHz) can be related to the difference in the Young's modulus used in the simulation (169 GPa) and as its real value.

Before coating, the resonance frequency of non-coated resonators (after functionalizing their surface with APTES) is measured and considered as the reference frequency (f_0) for all later frequency shift calculations ($\Delta f_{(i)} = f_{(i)} - f_0$). The notation $f_{(i)}$ refers to the resonance frequency of the sensor after coating of i -th polymer bilayer ($i = 4, 6, \dots, 12$). The chips are stored in nitrogen box for a few hours to be dried after each polymer

coating step. The polyelectrolyte coating is known to be not uniform up to 4 bilayers [81]. As a result, the frequency shift would not be linear below 4 bilayers and thus the first frequency shift is measured after coating of 4 bilayers. Thin polymer film successfully formed by the successive deposition of PAH and PSS up to 12 bilayers on the resonators surface. Five frequencies are recorded for each resonator after every 2 bilayers coating to calculate the error bar. Figure 3-26 shows the measured frequency shifts ($\Delta f_{(i)}$) of 34.81 MHz resonator versus the number of polymer coated bilayers and their corresponding masses. It can be seen that the frequency shift is linearly correlated with the number of coated polymer bilayers for all resonators.

Figure 3-26 Absolute frequency shift, $|\Delta f_{(i)}|$ versus number of coated PEM bilayers and its corresponding mass change for the 34.81 MHz resonator

The mass sensitivity of the resonator can be calculated now from the measured frequency shifts and Eq. (3-39). In order to obtain the limit of detection (LOD) and minimum

detectable mass (δm) of sensors, the standard deviation of the resonance frequency of each resonator is measured. For this purpose, the number of 30 data points ($n=30$) are recorded for each sensor with the rate of 1 sample/min. Frequency shift measurements are repeated for two more resonators with different lateral size and their measured resonance frequency, mass sensitivity, limit of detection and minimum detectable mass are listed in Table 3-7.

Table 3-7 Measured resonance frequency, mass sensitivity, noise, LOD and minimum detectable mass of three fabricated sensors

Frequency, f_0 (MHz)	Mass sensitivity, S_m ($\mu\text{m}^2/\text{ng}$)	Noise, 3σ (KHz)	LOD ($\text{ng}/\mu\text{m}^2$) ($\times 10^{-6}$)
34.81 ($L = 100\mu\text{m}$)	104.23	4.95	1.36
30.1 ($L = 120\mu\text{m}$)	106.31	6.24	1.95
24.2 ($L = 150\mu\text{m}$)	105.62	6.83	2.67

It can be seen from the Table 3-7 that the mass sensitivity is independent from the lateral size (L) of resonators as it expected from Eq. (3-39). The average S_m is $105.4 \mu\text{m}^2/\text{ng}$, which coincides with the theoretical mass sensitivity ($107.29 \mu\text{m}^2/\text{ng}$). This amount of mass sensitivity is higher than the commercialized mass sensors like QCM-D [82] and magnetoelastic [83] sensors with mass sensitivities of $1.13 \mu\text{m}^2/\text{ng}$ and $36.19 \mu\text{m}^2/\text{ng}$, respectively. The obtained mass sensitivity is also much higher than the piezoelectric membrane-based biosensor [84] with mass sensitivity of $16.5 \mu\text{m}^2/\text{ng}$. The main reason of this high mass sensitivity of the square resonator in comparison with mass sensors is its relatively thin resonator structure ($2 \mu\text{m}$).

3.6 Summary

In conclusion, a silicon Lamé-mode bulk acoustic resonator has been demonstrated for the use in mass sensing application. Mass sensitivity of $105.4\mu\text{m}^2/\text{ng}$, limit of detection of $1.36\times 10^{-6}\text{ ng}/\mu\text{m}^2$ and minimum detectable mass of 27.3 pg have been measured. The high mass sensitivity arises from miniaturization of the resonator (2 μm thick resonator) which leads to an increase in the relative frequency shift for a given amount of adsorbed mass. In addition, the compact size of the resonator helps that a small sample volume to be required in real bio applications. The demonstration of the sensor characterizations make this new technology suitable for the fabrication of compact, low cost and high performance biosensors platform for multiplexed detection.

Chapter 4

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4. Piezoelectric Aluminum Nitride (AlN) MEMS Resonator

This chapter describes a new design of piezoelectric aluminum nitride (AlN) resonator with checker-patterned electrode architecture for mass sensing application. The mass sensitivity of the developed sensor is analytically derived and confirmed with FEM simulations and experimental results. The main advantage of the designed AlN MEMS resonator is the very low power consumption which is useful in many applications like in vivo and network sensors.

4.1 Design and Simulation

A piezoelectric resonator is composed of a piezoelectric film sandwiched between two metal electrodes. The interdigitated (IDT) electrode fingers have been placed on a piezoelectric film that can excite acoustic waves within the piezoelectric layer. Several modes of vibrations can be designed either by adjusting the size and pitch of the electrode fingers or the resonator geometry and its anchoring points. In this study, a new piezoelectric AlN resonator is developed with checker-patterned electrode architecture. The developed resonator is actually an extension of the contour-mode resonator. The checker-mode resonator (CHR) consists of a square plate supported by four anchor beams attached at the corners. An array of interdigitated checker electrodes is used to simultaneously excite two-dimensional plate acoustic waves within the thin piezoelectric

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layer, one travelling along X-direction and the other along Y-direction (Figure 4-1(a)). It has 0.4 μm top Al, 1.0 μm AlN, 0.4 μm bottom Al along the thickness direction.

Figure 4-1 (a) Schematic view of the checker-mode resonator (0.4 μm top Al / 1.0 μm AlN / 0.4 μm bottom Al); (b) Three possible electrode configurations; top checker-patterned electrodes; top checker-patterned electrodes and full bottom electrode; checker-patterned on top and bottom

As mentioned above, resonance frequency can be changed by adjusting the size and pitch of the electrode fingers and hence resonators with multiple frequencies can be fabricated on a single wafer. Interdigitated electrode fingers can also be placed on either top and/or bottom metal layers, or only on top metal layer. Three possible electrode configurations are shown in Figure 4-1(b). For lateral field excitation (LFE), applied electric field is between adjacent electrodes within one layer [85, 86]. For the case of electric field applied across top and bottom electrodes, the resonator is considered under thickness field excitation (TFE). In this work, TFE configuration with top checker-patterned electrodes and full bottom electrode is considered. FEM simulation of the checker-mode resonator is carried out in ANSYS and the relative displacements in the main directions are shown in Figure 4-2. The material properties used in the simulations are listed in Table 4-1.

Figure 4-2 FEM simulations of the “checker-mode” in ANSYS and the relative displacements in the main directions

As shown in Figure 4-2, the checker-patterned electrodes can be used to simultaneously excite two intersecting acoustic waves, one travelling along X direction and the other along Y direction within a horizontal plane. The information of lines, spaces and checker gaps can be calculated back having the resonator dimensions and λ (Table 4-2).

In order to compare the checker-mode resonator characterizations with other piezoelectric vibrating modes, two other vibrating modes i.e. contour-mode resonator [87] and width-extensional mode resonator [85] are considered in the fabrication and test. Figure 4-3(a) shows a schematic diagram of an AlN contour-mode resonator shaped as a rectangular

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plate. For CMR, when an AC signal is applied across the thickness of the device, bulk acoustic waves are excited in the lateral plane and travel along resonator length.

Using different anchoring scheme on rectangular plate resonator, results in a width-extensional resonator (WXR). This mode is essentially based on excitation of longitudinal acoustic waves that extend and contract along the width of the resonator. Figure 4-3 (b) shows a schematic of an AlN WXR and direction that acoustic wave travels. All three explained resonators i.e. CHR, CMR and WXR are similarly composed of a 1.0 μm thick of AlN sandwiched between the two 0.4 μm thick Al electrodes in our design.

Figure 4-3 (a) Schematic of contour-mode resonator; (b) Schematic of the width-extensional mode resonator and acoustic wave direction

An ANSYS simulation is carried out to show the acoustic wave traveling direction and the mode of vibration of CM resonators (Figure 4-4). The figure shows only few of the sub-resonators as representatives of the overall device behavior.

Figure 4-4 2D FEM simulation in ANSYS, showing displacement in the cross section of contour-mode resonator (four parallel electrodes are considered here)

The resonance frequency of piezoelectric resonators can be obtained by the distance of the adjacent fingers in electrodes, λ and the resonator materials properties as shown in Eq. (4-1) [85]:

$$f_0 = \frac{1}{\lambda} \sqrt{\frac{E_{eq}}{\rho_{eq}}} \quad (4-1)$$

where ρ_{eq} is the equivalent materials density, and E_{eq} is the equivalent Young's modulus of the material stack that forms the resonator. The other two geometrical dimensions, length and thickness of the resonators can be independently designed to set the resonator electrical capacitance, C_0 , and its motional resistance, R_x . As the thickness of the electrodes is comparable to that of the piezoelectric film, it cannot be neglected. The below approximations are used to take the loading effects of the top and bottom metal electrodes into account in the resonance frequency of the checker-mode resonator:

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$$E_{eq} = \frac{E_{AlN}t_{AlN} + 1.5E_{Al}t_{Al}}{t_{AlN} + 1.5t_{Al}}, \quad (4-2)$$
$$\rho_{eq} = \frac{\rho_{AlN}t_{AlN} + 1.5\rho_{Al}t_{Al}}{t_{AlN} + 1.5t_{Al}}$$

where t_{Al} and t_{AlN} are aluminum and aluminum nitride thicknesses, respectively. Multiplier factor of ‘1.5’ (instead of 2) in Eq. (4-2) is due to the fact that the top checker-patterned electrode cover only half of the surface area. Material properties of Al, AlN and calculated equivalent properties of the resonators are listed in Table 4-1.

Table 4-1. Material properties values and calculated equivalent elastic modulus and density using Eq. (4-2) [88]

Material Properties	Value	Unit
Aluminium Elastic Modulus, E_{Al}	70	GPa
Aluminium Density, ρ_{Al}	2700	kg/m ³
Aluminium Nitride Elastic Modulus, E_{AlN}	350	GPa
Aluminium Nitride Density, ρ_{AlN}	3200	kg/m ³
Equivalent Young's Modulus, E_{eq}	226	GPa
Equivalent Materials Density, ρ_{eq}	3033	kg/m ³

Six different designs are selected for three piezoelectric resonators. Geometrical dimensions of these designs are listed in Table 4-2. Parameter λ is the distance of two adjacent fingers in one electrode. Using Eq. (4-1) and the equivalent properties between the resonator stacks from Table 4-1, the resonance frequency of the designed resonators are calculated and listed in Table 4-2. The obtained resonance frequencies from FEM simulations are also listed in this table.

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Table 4-2 Geometrical dimensions and obtained theoretical and simulated frequencies

	Design	L(μm)×W(μm)	λ (μm)	Theoretical Frequency, (MHz)	Simulated Frequency, (MHz)
1	CHR1	106×197	12	757.01	778.42
2	CHR2	154×154	76	119.53	120.25
3	CMR1	92×214	8	1135.51	1145.00
4	CMR2	100×100	38	239.05	243.25
3	WXR1	464×156	48	189.25	193.88
6	WXR2	408×118	40	227.10	236.94

4.2 Mass Sensitivity Derivation

The working principle of mass sensors is that the resonant frequency of the resonator decreases in response to a mass loading on its surface. For bulk acoustic resonator (BAW) mass sensors, the normalized mass sensitivity, S_M is defined with Eq. (3-36) [76, 89] which is repeated here for easy reference:

$$S_M = - \lim_{\delta m \rightarrow 0} \frac{1}{f_0} \left(\frac{\delta f}{\delta m} \right) \quad (4-3)$$

where $\delta m = \rho_d t_d$ is the uniform external added mass on the surface per unit area of the sensor, due to absorption or thin film deposition; ρ_d and t_d are the density and thickness of deposited mass and $\delta f = f - f_0$ is the frequency shift in response to the mass loading. The negative sign in Eq. (4-3) is to be cancelled out with frequency change (δf) since the resonance frequency decreases after adding the mass on the resonator surface.

In the checker-mode resonator, the velocity of each point on the resonator is not uniform over its entire width and length since the mode-shape of vibration obeys a harmonic motion in x and y directions. Indeed the mass loading is not distributed where the kinetic

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energy is maximum in this mode. This fact can be taken into account by computing the effective deposited mass at the points with maximum velocity. A cross-section of the checker-mode resonator and the effective mass are shown in Figure 4-5.

Figure 4-5 Acoustic wave in resonators cross section and replacing the total deposited mass (δm) with effective deposited mass (δm_{eff}) in the points with maximum velocity

The harmonic velocity in x and y directions can be described as below:

$$\begin{cases} v_x = V_m \cdot \sin\left(\frac{2\pi}{\lambda} x\right) \\ v_y = V_m \cdot \sin\left(\frac{2\pi}{\lambda} y\right) \end{cases} \quad (4-4)$$

where V_m is the maximum velocity amplitude in both x and y directions. As a result, the effective deposited mass can be obtained from the total mechanical kinetic energy (KE_{tot})

in the range between two points with zero velocities (between $-\frac{\lambda}{4}$ and $\frac{\lambda}{4}$), as below:

$$\begin{aligned}
 \delta m_{eff} &= \frac{KE_{tot}}{\frac{1}{2}(V_m^2 + V_m^2)} = \frac{2}{(V_m^2 + V_m^2)} \iint_{-\frac{\lambda}{4}}^{\frac{\lambda}{4}} \frac{1}{2} (v_x^2 + v_y^2) (\rho_d t_d) dx dy \\
 &= \rho_d t_d \iint_{-\frac{\lambda}{4}}^{\frac{\lambda}{4}} \left(\sin^2 \left(\frac{2\pi}{\lambda} x \right) + \sin^2 \left(\frac{2\pi}{\lambda} y \right) \right) dx dy = \frac{\delta m}{2}
 \end{aligned} \tag{4-5}$$

where δm_{eff} is the effective sensed mass and δm is the total deposited mass. Equation (4-5) indicates that only half of the loading mass is actually sensed by the resonator. As indicated by the Sauerbrey equation [76], the change in resonance frequency is linearly related to the effective mass of a material deposited on a surface.

$$\frac{\delta f}{f_0} \approx - \frac{\rho_d t_d}{\rho_s t_s} = - \frac{\delta m_{eff}}{m_s} \tag{4-6}$$

where ρ_s and t_s are the density and thickness of the resonator and m_s is the resonator mass per unit area. Substituting the effective sensed mass, δm_{eff} with the total deposited mass, δm and replacing the result in Eq.(4-3), the effective mass sensitivity of the checker-mode resonator obtained as below:

$$S_M = \frac{1}{2m_s} = \frac{1}{2\rho_s t_s} \tag{4-7}$$

where ρ_s is the resonator mass per unit area of the resonator and can be replaced with ρ_{eq} in Table 4-1. The obtained relation for mass sensitivity of the checker-mode resonator is also similar to what is obtained for CM and WX resonators in [90].

It can be inferred from Eq. (4-7) that the mass sensitivity enhances with increasing the frequency of operation and decreasing the resonator thickness. The resonance frequency of designed resonator is lithographically set and can be independently designed from the

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resonator thickness. Film thickness of the resonator structure is an important parameter to set the device mass sensitivity. In this work, the mass sensitivity is improved with increasing the resonance frequencies of the devices, without compromising the fragility of the resonator by decreasing the thickness.

The mass sensitivities of CHR1 (757 MHz) and CMR1 (1135 MHz) designs are analytically evaluated using Eq. (4-7) and found to be 1284.81 Hz.cm²/ng and 1776.75 Hz.cm²/ng, respectively. A series of 2D and 3D Finite Element Method (FEM) analyses has been performed with the aid of ANSYS to validate the obtained analytical values of mass sensitivity (S_M). A thin square-shaped mass is added to the surface of each resonator (Figure 4-6). Then, the device sensitivity is evaluated by gradually varying the density of the added mass and computing the corresponding linear shift in the resonance frequency. The material properties of Al and AlN used in these simulations can be found in Table 4-1. The Young's modulus and Poisson ratio of the deposited layer are considered equal to 300 MPa and 0.5 respectively in accordance to the properties of polyelectrolyte multilayers (PEM) [80, 91] used in Section 4.4.1.

The resonance frequencies of CMR1 are simulated for different coated layer thicknesses (different densities here) and the result is depicted in Figure 4-7.

Figure 4-7 Frequency of CMR1 ($92\mu\text{m}\times 214\mu\text{m}$, with $\lambda=8\text{ }\mu\text{m}$) after coating different thickness of polymer ($f_0=1145.28\text{ MHz}$)

As it can be seen the non-linear behavior of the above resonator starts after coating of about $1.5\text{ }\mu\text{m}$ of polymer. The mass sensitivities of CHR1 and CMR1 designs are calculated to be $1285.7\text{ Hz.cm}^2/\text{ng}$ and $1778.3\text{ Hz.cm}^2/\text{ng}$, respectively which shows a good match between analytical and simulated values.

The sensor resolution of mass sensors is determined by Limit of Detection (LOD). LOD describes the smallest amount of mass that a sensor can detect considering the existing noise. The uncertainty in the measured frequencies can be caused by device imperfection and the electronics device and environment noise. Smaller amount of LOD means that smaller mass can be detected. The LOD of an acoustic mass sensor is given by [43]:

$$LOD = \frac{N}{S_M} \quad (4-8)$$

where N is the noise level of the sensor and is equal to 3 times of the standard deviation of measured resonance frequency of a bare resonator ($N = 3\sigma$). The mass sensitivity and LOD of the designed sensors are experimentally measured in section 4.4.

4.3 Fabrication Process Flow

The layouts of the designed resonators were made in Coventorware software. The layouts of WXR1 resonator (0.4 μm Al / 2 μm AlN / 0.4 μm Al) and CHR2 resonator (0.4 μm Al / 2 μm AlN / 0.4 μm Al) are shown in Figure 4-10 and Figure 4-11.

Figure 4-8 Layout of WXR1 resonator (diaphragm size 464 μm x 156 μm)

Figure 4-9 Layout of checker-mode resonator (CHR2) (diaphragm size 154 μm x 154 μm)

The fabrication process of resonators started with a silicon wafer with 0.3 μm thermal oxide, 400 nm of bottom Al and 1 μm AlN film on top. AlN thin film deposition was done for (002) crystal orientation and the film was verified to have a good texture. The first mask was used to pattern the AlN layer and was etched with $\text{Cl}_2/\text{BCl}_3/\text{Ar}$ chemistry using SiO_2 hard mask. The bottom Al metal was patterned and etched with second mask for ground electrode contact. Then, additional SiO_2 layer was deposited as the isolation between top and bottom electrodes and patterned with third mask. The 400 nm top Al metal was deposited by physical vapor deposition (PVD) and patterned with the fourth mask. Then 0.3 μm SiO_2 layer below the bottom Al metal was etched with the fifth mask. Finally, the device was released by isotropic etching of Si substrate in XeF_2 chamber. Fabrication process flow is summarized in Figure 4-10.

Figure 4-10 The fabrication process steps: (a) Started with a silicon wafer with thin layer of thermal oxide, 400 nm of bottom Al and 1 μm of AlN film on the top; (b) Pattern and etch the AlN layer using SiO_2 hard mask, Open vias in AlN to access bottom electrode; (c) Protect the opened vias with photoresist, Pattern and etch the bottom Al layer; (d) Protect AlN and bottom Al using SiO_2 , Pattern and etch the SiO_2 (e) Sputter 400 nm Al on top, Pattern and etch the top electrodes; (f) Pattern and Etch the bottom SiO_2 , XeF_2 dry release of the AlN resonator.

SEM images of released resonators (CM, WX and CH) are shown in Figure 4-11.

Figure 4-11 SEM images of the released resonators (a) Contour-mode resonator (CM); (b) Extensional mode resonator (WX); (c) Checker-mode resonator with 6 electrodes pads (CH)

4.4 Sensor Characterizations and Analysis

In order to characterize the fabricated resonators, its surface area should be coated with a controlled mass. In this work, polyelectrolyte multilayer (PEM) is used for this purpose [77, 78]. After coating a specified amount of polymer, the resonance frequency shift and the new frequency are measured after each layer coating. The details of the required steps for the sensor characterization are given in the following sub sections.

4.4.1 Polyelectrolyte Multilayer Coating

The required steps for polymer coating are similar to Section 3.4.1 except the first step. Aluminum surface of the resonator is naturally oxidized in the atmosphere and forms a thin layer of aluminum oxide (Al_2O_3). Therefore, the resonator surfaces are negatively charged and PEM can be directly coated on their surfaces (no need to charge the surface with APTES as in silicon). The required steps for polymer coating on aluminum surface are summarized in Figure 4-12.

Figure 4-12 Required steps for PEM coating: (1) Dipping in PAH solution; (2) Rinsing in DI water; (3) Dipping in PAH solution; (4) Rinsing in DI water.

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The remained steps for polymer coating are similar to the steps for polymer coating on silicon which is fully explained in section 3.4. The thickness and the mass per unit area of the coated polymer bilayers can also be estimated using the method explained in section 3.4.1.

4.4.2 Experimental Results and Discussion

Agilent E8364B network analyzer and probe station were used to measure the resonance frequency of the resonators. All the measurements were conducted in the atmospheric pressure and temperature of 22 °C. The frequency spectrums of CHR1 resonator with all other adjacent resonance modes are shown in Figure 4-13.

Figure 4-13 Frequency spectrum of CHR1 at -10 dBm measured using Agilent E8364B network analyzer and probe station ($f_0 = 770.11$ MHz)

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As it can be seen from Figure 4-13, the peak related to this mode (Checker-mode) is clearly raised. The measured Q-factor corresponding to this mode is 267. The resonant frequency and phase shift of CHR2 is also measured and shown in Figure 4-14.

Figure 4-14 The measured resonant frequency and phase of CHR2 at -10 dBm ($f_0 = 119.78$ MHz)

The measured Q-factor of this mode is 870. The resonance frequencies and phase shifts of CMRs are also measured. The resonance frequencies of CMR1 and CMR2 are 1113.33 MHz (Figure 4-15) and 238.08 MHz (Figure 4-16), respectively. The measured Q-factors of these modes are 603 and 550, respectively.

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Figure 4-15 The measured resonant frequency and phase of CMR1 at -10 dBm ($f_0 = 1112.40$ MHz)

Figure 4-16 The measured resonant frequency and phase of CMR2 at -10 dBm ($f_0 = 237.81$ MHz)

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The resonant frequencies and phase shifts of WXR1 and WXR2 are also measured and shown in Figure 4-17 and Figure 4-18. The resonance frequencies of WXR1 and WXR2 are 151.54 MHz and 237.046 MHz and their Q-factors in the air are 551 and, 281 respectively.

Figure 4-17 The measured resonant frequency and phase of WXR1 at -10 dBm ($f_0=151.77$ MHz)

Figure 4-18 The measured resonant frequency and phase of WXR2 at -10 dBm ($f_0 = 237.46$ MHz)

After doing the measurements in the air, the Q-factor of the resonators were measured in the liquid to observe the feasibility of using these resonators in biosensing application. With the RF probe still in contact with the device's pads, one droplet of a high impedance liquid (Fluorinert) with a density of 1.94 g/cm^3 and a kinematic viscosity of $14.0 \text{ mm}^2/\text{sec}$ was dispensed onto the surface of the device. The Q-factor of all resonators drops intensively as a result of the liquid. For instance, the resonant frequency of the WXR2 in Fluorinert was 237.04 MHz (Figure 4-18) with Q-factor of 281 in the air. The frequency shifted 2.183 MHz in Fluorinert and Q-factor reduced to 29, which attenuated 90% compared to the Q in air (Figure 4-19).

Figure 4-19 Resonant frequency and phase of WXR2 resonator in Fluorinert at -10 dBm, the Q-factor attenuated 90% compared to the Q in air

In order to study the relationship between the added mass and frequency shift of resonators, the PEM bilayers were used in layer-by-layer coating manner. Before coating the polymer, the resonance frequency of non-coated resonators was measured and considered as the reference frequency (f_0) for all later frequency shift calculations ($\delta f_{(i)} = f_{(i)} - f_0$) for each resonator. The notation $f_{(i)}$ refers to the resonance frequency of the sensors after coating of i -th polymer bilayer ($i = 4, 6, \dots, 12$). The coated polymer bilayer is known to be non-uniform up to 4 bilayers [81]. As a result, the frequency shift would not be linear below 4 bilayers and the first frequency measurement is carried out only after coating of 4 bilayers. Then, a new 2 polymer bilayers were coated on the chip and corresponding frequency shifts are recorded. The process of coating and measurement continued up to 12 bilayers by 2 bilayers intervals

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for each measurement. For each measurement, 5 frequencies were recorded for each resonator to calculate the error bar. Figure 4-20 shows the measured resonance frequency ($f_{(i)}$) of checker-mode resonator after coating of different number of polymer bilayers.

Figure 4-20 The measured resonance frequency of checker-mode resonator after coating different number of PEM bilayers

Due to the mass-sensitive nature of the piezoelectric sensor, the resonance frequency of checker-mode resonator obviously decreases after deposition of masses. Similar frequency measurements were performed for all other fabricated resonators (CHR1, CMR1, CMR2, WXR1 and WXR2) and the frequency shifts were obtained after coating different number of polymer bilayers. Figure 4-21 shows the measured frequency shifts ($\delta f_{(i)}$) of resonators versus the number of coated polymer bilayers. It can be clearly seen that the frequency shift is linearly correlated with the number of coated polymer bilayers for all resonators. For instance, the regression equation of CMR1 is as below:

$$y = 0.417x - 0.715, \text{ Correlation coefficient} = 0.995$$

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It should be noted that line fits have to extrapolate to zero frequency shift at zero mass. The line fits in Figure 4-21 pass from the neighborhood of zero, as there are measurement noises.

Figure 4-21 Absolute frequency shift versus number of deposited polymer bilayers (R^2 : correlation coefficient)

Dividing the resonance frequency shift by the value of the adsorbed mass per unit surface area of the correspondent resonator, the mass sensitivity of the devices are calculated (Eq.(4-3)) and listed in Table 4-3. The mass sensitivity (S_m) of the resonators is calculated based on the Eq. (3-36) to compare with the mass sensitivity of the Lamé-mode resonator of chapter 3.

Table 4-3 Measured resonance frequency, frequency shifts and mass sensitivity of fabricated resonators after 12 bilayers coating.

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Device	Frequency, f_0 (MHz)	Δf (MHz) after 4 bilayers	ppm change of freq after 4 bilayers coating	Mass sensitivity, S_M , ($\mu\text{m}^2/\text{ng}$)
CHR1	773.42	-2.38	-3079.68	175.42
CHR2	120.01	-0.09	-740.82	167.41
CMR1	1112.41	-1.61	-1444.62	152.37
CMR2	233.33	-0.46	-1955.36	168.55
WXR1	151.85	-0.21	-1383.99	155.56
WXR2	237.82	-0.27	-1149.77	161.49

The obtained experimental results show higher mass sensitivities for the checker mode resonators compared to the other two modes (CMR and WXR). In addition, comparison between the experimental values of mass sensitivity with the analytical values of $161.36 \mu\text{m}^2/\text{ng}$ for CHR1 and $151.65 \mu\text{m}^2/\text{ng}$ for CMR1 (refer to Section 3) shows very strong coherence between theory and experiments and further confirms a clear understanding of the sensor operations.

These achieved mass sensitivities are comparable with many state of the art mass sensors like QCM [40], SAW [12] and FBAR [20-21, 41] sensors. For instance, the mass sensitivity of $175 \mu\text{m}^2/\text{ng}$ of CHR1 is more than 4 times higher than that of a recently reported QCM with mass sensitivity of $39.4 \mu\text{m}^2/\text{ng}$ [3] and about 3 times higher than that of a similar AlN resonator with mass sensitivity of $53.56 \mu\text{m}^2/\text{ng}$ [26]. Although the obtained results are not the best reported mass sensitivities in the literature, they are still comparable with recent reported resonators. For instance, a 800 MHz FBAR sensor with mass sensitivity of $250 \mu\text{m}^2/\text{ng}$ and a minimum detectable mass of about $1 \text{ ng}/\text{cm}^2$ is reported in [5].

The limits of detection (LOD) of three selected resonators were measured and listed in Table 4-4. In order to evaluate the total noise (N), the resonance frequencies of resonators

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were measured during the time and number of 30 data points ($n=30$) are recorded for each sensor with the rate of 1 sample/min.

Table 4-4 Limit of detection of CM, CH and WX resonators

Device	Noise, 3σ (KHz)	Limit of Detection LOD (ng/cm ²)
CMR1	37.04	18.7
CHR1	9.97	24.1
WXR2	3.60	14.9

LOD describes is the smallest amount of mass per unit area which produces a measurable output signal considering the existing noises. As the experiments are performed in normal environment, the obtained LOD includes the ambient noise, measurement equipment noise and the resonator structure noises which are all considered as measurement limitations. As a result, the obtained LOD is relatively high comparing the current sensors (For instance QCM has the limit of detection of 5 ng/ $\cdot$ cm² [96], LOD of the developed sensors can be improved further with optimizing the electronic interface in future work.

Having a resonator structure with high mass sensitivity and a very small limit of detection is useful and can be used in many different areas. However, in order to have a high resolution sensor, it is not only necessary to improve their physical structure, but it is also essential to deal with the chemical properties of the sensitive layer responsible for the adsorption process.

4.5 Summary

In this chapter, design, fabrication, and test of new AlN MEMS based resonators have been demonstrated. The experimental results have shown that the developed mass sensors

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have high potential to be exploited as gravimetric mass sensors. A new AlN resonator (CHR) is introduced, and high mass sensitivity of $175.42 \mu\text{m}^2/\text{ng}$ with detection limit of $18.7 \text{ ng}/\text{cm}^2$ is achieved for a 773 MHz resonator. The obtained high mass sensitivity arises from small resonator thickness. In addition, due to the compact size of the resonator, only a small sample volume is required in real bio applications. Furthermore, the designed AlN MEMS resonator has a very low power consumption which is useful in many applications like in vivo and network sensors. The demonstration of the sensor characterizations makes this new technology suitable for the fabrication of compact, low cost, and high performance biosensors platform for multiplexed detection.

Chapter 5

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5. Biochemical Sensing

In this chapter, the Lamé-mode bulk acoustic resonator; which were designed and fabricated in chapter 3; is characterized for bio sensing and detecting the binding assays. Specific protein binding interactions using the well characterized biotin/streptavidin system is performed to verify the performance of the sensor with bio materials. The resonant frequency of the biotin modified resonator is measured to decrease by 315 ppm when exposed to streptavidin solution for 15 minutes with a concentration of higher than 10^{-7} M, corresponding to an added mass of 3.43 ng on the resonator surface. An additional control is added by exposing a BSA covered device to streptavidin in absence of the attached biotin. No resonance frequency shift is observed in this case, confirming the specificity of the detection. The sensor-to-sensor variability is also measured to be 4.3%. Consequently, the biotin modified developed sensor can be used to observe biotin-streptavidin interaction without the use of labelling or molecular tags. The developed sensor can be used in a variety of protein-ligand systems and also high throughput screening for drug discovery.

Although the chemical/biochemical sensor market growth rate is relatively high, the majority of chemical analyses are still dependent on costly bench-top instruments laboratory analyses. They are highly sensitive and selective, but the analyses are performed off-line and typically time and labor intensive, thus limiting their applications. Therefore, inexpensive, highly sensitive chemical/biochemical sensors with ability of in-

place measurement are still in great demand [5, 97]. From this point of view, micromachined chemical/biochemical sensors, which make use of well-established semiconductor fabrication processes, are one of the most promising candidates [97, 98]. They provide an interesting means to analyze the content of a biological sample based on the direct conversion of a biological event to an electronic signal and allow highly sensitive and selective detection of micro-organisms or toxins. Compared to other competing technologies, low power consumption, small sensor and sample volumes (e.g. blood), fast response times, excellent manufacturing/process repeatability and mass production capability with high production yield are strong advantages of microsensor technology.

Micro electro-mechanical biosensors allow limited uses or show some promises for label-free study of biomolecular interactions: surface vibration spectroscopy [99], optical methods [20, 99] mass sensitive quartz-crystal-microbalances QCMs [100, 101], Film Bulk Acoustic Resonator (FBAR) [55, 102] and microcantilevers [22, 23].

In order to characterize the fabricated resonator, the bio entities are immobilized using a water droplets on the resonator. The droplets volume of 0.3 nL is spotted on the centre of the resonator top surface, and the resonator response is measured in liquid environment. The resonator silicon surface is activated by 2% 3-aminopropyltriethoxysilane (APTES). When the target species (streptavidin) interacts or binds with the surface bound biotin, the mass load of the resonator is increased and the resonant frequency proportionally decreased.

5.1 Droplet Coating on the Sensor Surface

The structure of the resonator was fully explained in chapter 3. In the remainder of this chapter, the mass sensitivity of the developed sensor is estimated in the aqueous environment. For this purpose, a polymer multilayer was used as a controllable mass on the resonator surface. The experiment performed both in the air and water. Then the mass sensitivity was estimated using biotin-streptavidin in the aqueous environment.

In order to functionalize the sensor for bio-experiments, the resonator surface should be spotted with a water droplet. As the viscous damping increases when the droplet contacts with the surface, both frequency and Q-factor will be affected. The maximum droplet volume that can be applied is assumed to be the droplet volume that has a water contact area of approximately $\pi L^2/4$ (i.e. 78% of the top surface area) which is $\pi L^3/12$. For the resonator with side length of 100 μm , the droplet volume is approximately 0.3 nL. The water droplets were spotted on the centre of the resonators using a KRÜSS-DSA100 contact angle measurement instrument. As the minimum droplet size of this machine is 1.5 μL , to get the right volume, time has to be passed for evaporation. The evaporation time for a 1.5 μL water droplet to dry out completely is more than 30 minutes. As a result, the desired droplet in the resonator centre can be obtained before this time. Figure 5-1 shows a schematic of the resonator and water droplet spotted on the center of the resonator far from the surrounding electrodes.

Figure 5-1 Schematic of the resonator and water droplet spotted on the centre of the resonator far from the surrounding electrodes. The maximum water droplet volume that can be applied to each resonator is assumed to have covered 78% of the top surface from optical inspection.

The frequency response and Q-factor of the resonators after spotting the water droplet were measured. The resonant frequency and extracted Q-factor for the measured data are 32.42 MHz and 280, respectively. As expected the Q-factor of the resonator extremely decreased after spotting the water droplet on its surface as viscous damping in water is much higher than what is in the air.

5.2 Mass Sensitivity Estimation Using PEM in the Water

The mass sensitivity of the micro machined silicon sensor in air using sequential deposition of Polyelectrolyte Multilayer (PEM) is estimated in section 3-5. In this section, the same PEM coating steps were used, but the measurements were carried out under the water droplet (instead of air). The chips are attached to the same custom-made printed circuit board (PCB) and the measurements are performed with an Agilent 4395A network analyzer. The applied driven power by network analyzer is -10 dBm (70 mV @ 50Ω).

Similar to chapter 3, the resonance frequency of non-coated resonators (after functionalizing their surface with APTES) was measured and considered as the reference frequency (f_0) for all later frequency shift calculations. Ten frequencies were recorded for each resonator after every 2 bilayers coating to calculate the error bar. Figure 5-2 shows the measured frequency shifts ($\Delta f_{(i)}$) in air and water for a $100\ \mu\text{m}$ resonator length versus the number of polymer coated bilayers and their corresponding masses. It can be seen that the frequency shift is linearly correlated with the number of coated polymer bilayers for all resonators.

Figure 5-2 Absolute frequency shift, $|\Delta f_{(i)}|$ versus number of coated PEM bilayers and its corresponding mass change for the resonator with length of $100\ \mu\text{m}$ in air and water

Frequency shift in the liquid environment have larger error bar comparing to the air as it is harder to read the exact resonance frequency because of lower Q-factor. The obtained mass sensitivity from both air and water are calculated and compared. The average S_m in the air was $105.4\ \mu\text{m}^2/\text{ng}$, which was too close to the theoretical mass sensitivity

($107.29 \mu\text{m}^2/\text{ng}$). However, the average calculated mass sensitivity of the resonator with the water droplet is reduced to $79.53 \mu\text{m}^2/\text{ng}$. The main reason of the difference between the obtained mass sensitivities in air and water is the absolute frequency shift of the resonators in the water. A more careful and systematic analyses are required to explain the effects in the aqueous environment.

5.3 Mass Sensitivity Estimation Using Biotin-Streptavidin

One common strategy to characterize the biosensors is to take advantage of the strong interaction between biotin-streptavidin which has one of the highest non-covalent affinities known with a $K_d \approx 10^{-14}$ – 10^{-16} M [103, 104]. The wide range of applications makes biotinylated sensor surfaces attractive for almost all surface based sensing techniques and the aim of this study is also to characterize our developed sensor for these applications. The signal transduction process in the Lamé-mode resonator is due to added mass resulting from interaction of the immobilized probe biotin and target species streptavidin causing the device's resonant frequency to decrease.

5.3.1 Materials and Reagents

The 3-aminopropyltriethoxysilane (APTES) used to modify the silicon resonator surface, is purchased from Sigma–Aldrich Chemie GmbH, Germany. Biotin (1 mg/mL) and streptavidin are purchased from Pierce, Rockford, USA. The blocking reagent, Bovine Serum Albumin (BSA), is purchased from Sigma–Aldrich Chemie GmbH, Germany. Phosphate Buffered Saline (PBS) used to wash the non-specific binding is purchased from Invitrogen, USA. All other chemical reagents used are of analytical reagent grade.

The deionized (DI) water used in all experiments is produced by a water purification system, TKA, from Millipore Corporation, USA.

5.3.2 Immobilization Steps for Biotin-Streptavidin on Silicon Resonator

For the immobilization of the biotin-streptavidin on the surfaces of the resonator microstructures, the below steps were taken:

5.3.2.1 *Cleaning the silicon surface*

- 1- Put the chips in 95% ethanol (500 μ L ultrapure water +9.5 ml Ethanol)
- 2- Put the petri dish contacting the chip in the shaker for 5 minutes
- 3- Plasma cleaning with oxygen (Power 100 W, Time 60 Sec, Gas: O₂)

5.3.2.2 *Using aminopropyltriethoxysilane (APTES) for surface fictionalization*

APTES is used to fictionalize the silicon surface. The 2% APTES should be prepared with mixing the following solutions: (i) 2% APTES, 99% (200 μ L); (ii) 95% Ethanol (9.5 mL); (iii) Ultrapure water (500 μ L). After preparing the solution with mixing the below steps should be taken for APTES coating:

- 1- Drop the solution onto the resonator and incubate it in the fume hood at room temperature for 2 hours
- 2- Wash non-specific binding of the APTES using the Ethanol (3 times, every time 5 minutes in the shaker)
- 3- Cure the chips in the oven with 120°C for 15 minutes

5.3.2.3 *Biotin immobilization:*

In order to immobilize the biotin on the functionalized silicon surface, the below steps should be taken:

- 1- Take the 2 mg of biotin out of refrigerator and let it to equilibrate with room temperature before opening
- 2- Add small amount of ultrapure water (e.g. 100 μL) to dissolve the concentrated biotin. Mix the biotin well using the pipette up and down
- 3- Mix the above solution with 2 mL ultra pure water and shake it with vortex maker. The obtained solution is 1 mg/mL solution of biotin
- 4- Incubate the chips in above solution in room temperature for 1 hour
- 5- Wash the chips with ultrapure water 3 times, each time for 5 minutes

5.3.2.4 Streptavidin immobilization:

In order to immobilize the streptavidin on the biotin-modified surface, the below steps should be taken:

- 1- Prepare streptavidin with desired concentration (10^{-15} M- 10^{-5} M in our case).
- 2- Incubate the chips in the streptavidin solution (with different concentration) for 15 minutes.
- 3- Wash the chips with Phosphate Buffered Saline (PBS), 3 times, each time for 5 minutes.
- 4- Wash the chips with ultrapure water.
- 5- Dry the chips in the nitrogen.

5.3.3 Measurement Results and Discussion

Four identical resonators (with the length of 100 μm) are prepared. The resonator surfaces are firstly cleaned using oxygen plasma for 60 sec. This step also promotes the availability of surface hydroxyls for reaction with the silanol group in the subsequent step. In the next step, the silicon resonator surfaces are modified with a 2% solution of 3-aminopropyltriethoxysilane (APTES) for 2 hours similar to the process done in the section 3. After silanization, unbound silane is removed by three washes with ethanol.

APTES-treated chips are functionalized with biotin with incubating the resonators in 1 mg/mL solution for 1 hour. One of the resonators is selected as the reference and its surface is blocked with Bovine Serum Albumin (BSA). In order to measure the resonator frequency shifts, the resonator are completely dried in nitrogen first. Then, as the resonance frequency in the aqueous environment is interested, a water droplet is spotted on the resonator top surface as described in the previous section. Then, the resonance frequency of the resonators are measured and considered as the reference frequencies (f_0) for that specific resonator.

The next step is to apply the streptavidin on the biotin modified resonator. Before starting the frequency shift measurement, the biotin–streptavidin reaction is confirmed through fluorescent microscopy with streptavidin molecules labelled with fluorophore. The fluorescent microscopy confirmed that 5 min after the introduction of the streptavidin solution, the interaction will be occurred and a strong fluorescent signal is obtain (Figure 5-3). In fact, the biotin–streptavidin binding needs only a few seconds to occur because of high affinity interaction.

Figure 5-3 Flourescent micoscopy images of the resonators after streptavidin immobilization. The resonators are coated with different streptavidin concentrations and imaged separately using a same combination of excitation and emission filters.

The next step is that to expose the sensors to different streptavidin concentration in the range of 1.88×10^{-15} M (100 fg/mL) to 1.88×10^{-5} M (1 mg/mL). Chips are only kept for 15 minutes in streptavidin solution to bind with biotin.

The open-loop frequency response of the resonators is measured at atmospheric pressure and room temperature after wire bonding, and measured again after immobilization to estimate the mass loading by the immobilized streptavidin. All bonding wires and pads are sealed with UV (ultra violet) curable epoxy and cured under UV light for 1 hour. The silicon chip mounted on a Quad Flat Package (QFP16) is shown in Figure 5-4.

(c)

Figure 5-4 (a) Schematic of wire-bonding of the resonator to the QFP16 (b) Cross-section of the wire-bonded silicon chip (c) Custom-made PCB for measuring the resonance frequency

Agilent 4395A network analyzer is used to measure the resonance frequency signal. Figure 5-5 shows the fully differential drive-and-sense measurement setup and the overall measurement setup implemented in this work. The electrical elements are placed on a printed circuit board (PCB). The designed PCB layout in ALTIUM DESIGNER software is attached in Appendix B.

Figure 5-5 The overall measurement setup implemented in this work

After immobilization of different streptavidin concentrations, the chips are completely dried and a water droplet spotted in the resonator's center for the measurement purpose as described in section 5.1. Then, the new resonance frequencies are measured after exposing of each streptavidin concentrations. The streptavidin fraction in the solution and corresponding sensor frequency shifts are shown in Figure 5-6.

Figure 5-6 The streptavidin fraction in the solution and corresponding sensor frequency shifts after 15 mins of interaction with biotin. The standard deviation in these experiments shown is based on many replicates of the frequency measurements after bonding the streptavidin with coated biotin

As it can be seen in Figure 5-6, the curve rises linearly while the available binding sites are being populated and begins to saturate when fewer sites are left available. The dynamic range of the sensor is found between the concentrations of 10^{-13} M- 10^{-7} M. The detection limit of the streptavidin sensor is estimated to be 10^{-13} M as the frequency shift is not considerable below this concentration. The frequency shift reaches to a plateau after putting the biotin-modified chip in streptavidin solution of 10^{-7} M and the saturation occurring. The reference sensor (the sensor which is blocked with 1 mg/ml BSA) shows a slight frequency shift with increasing analyte concentration with a maximum shift of about 70 Hz at the largest concentration. This shows that the resonator surface had been successfully blocked by BSA pre-treatment.

The resonance frequency of the resonator after saturation is 31.12 MHz that shows a 315 ppm drop comparing with the resonance frequency of the sensor before exposing to the streptavidin. The mass sensitivity of the sensor can now be calculated from the frequency shift at the saturation and the total deposited mass on the sensor surface. As the streptavidin estimated surface footprint is $4 \text{ nm} \times 5 \text{ nm}$ [105], the surface density of it in the saturation is $5 \times 10^4 \text{ molecules}/\mu\text{m}^2$. Given that streptavidin possess molecular weights of 53,000 Da (mg/Mol), the added mass per unit area is $4.4 \text{ fg}/\mu\text{m}^2$. This estimated deposited mass is close to the real measured mass per unit area on the FBAR surface, $3.6 \text{ fg}/\mu\text{m}^2$ [106]. The sensor has an active resonating area of $7.8 \times 10^5 \mu\text{m}^2$, so the actual mass increase on the sensor active area due to the biotin-streptavidin interaction is about 3.43 ng. Consequently, the mass sensitivity of the sensor is obtained with replacing the deposited mass per unit area and its corresponding frequency shift in Eq. (3-36). The average mass sensitivity of the sensor in the dynamic range (10^{-13} M - 10^{-7} M), is obtained to be $87.76 \mu\text{m}^2/\text{ng}$. The calculated mass sensitivity is in a good agreement with result obtained from the characterization of the sensor with PEM in water ($79.53 \mu\text{m}^2/\text{ng}$) in previous section. The results of the sensor mass sensitivity are summarized in Table 5-1:

Table 5-1 Measured resonance frequency and mass sensitivity of the Lamé-mode sensor coated with PEM and Biotin-Streptavidin binding in air and water

Resonator Added Mass and Test Environment	Mass sensitivity, S_m , ($\mu\text{m}^2/\text{ng}$)	Q-factor
Bare Resonator, Air	-	1800
Bare Resonator, Water	-	280
PEM Coated Resonator, Air	104.23	1230
PEM Coated Resonator, Water	79.53	225
Biotin-Streptavidin Coated Resonator, Water	87.76	208

The repeatability of these data is investigated with repeating the above experiment for two similar sensors of the same chip. The device-to-device variance in the frequency shift is recorded to be 500 Hz, corresponding to 4.3% change in the mean of the frequency shift.

5.4 Summary

The capacitive detection of the biotin–streptavidin binding with the mass sensitivity of $87.76 \mu\text{m}^2/\text{ng}$ in aqueous environment is demonstrated. Contrary to the common method wherein sensor surfaces are coated by a thin gold layer for the immobilization of probe molecules, the immobilization of biotin molecules took place directly on the silicon surface using APTES. A significant frequency change was observed when the interaction takes place on the surface of biotin functionalized resonators as opposed to minor frequency change of unfuctionalized (BSA blocked) sensor. Due to the small size of the resonator they can be mass produced and arrayed for analysis of multiple targets simultaneously. These results encourage the efforts on using Lamé-mode resonator for fast and label free analysis of biomolecular interactions.

Chapter 6

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6. Conclusion and Future Work

6.1 Conclusion

The main objective of this research is the performance improvement of micro electromechanical resonators in chemical and biological mass sensing applications. Two particular resonator types are designed, fabricated and their mass sensitivities characteristics are studied in both gaseous and liquid environments. The first designed resonator is a single-crystal silicon squared resonator with dielectric filled capacitive excitation mechanism. This resonator is fabricated using the silicon-on-insulator MEMS process. In order to study the mass sensitivity of the fabricated resonator, a polyelectrolyte multilayer (PEM) is used to coat on the resonator surface. The resonator has the resonance frequency of 34.81 MHz and mass sensitivity of $105.4 \mu\text{m}^2/\text{ng}$ and the limit of detection of $1.36 \times 10^{-6} \text{ ng}/\mu\text{m}^2$. Experimental measurement results of the mass sensitivities agree with theoretical predictions, especially in the air. The developed sensor shows a high potential to be used in bio detection applications compared to previous mass sensors in the literature (Chapter 3).

The second resonator is a newly designed piezoelectric AlN resonator with checker-patterned electrode architecture. The mass sensitivity of the developed sensor is analytically derived and confirmed with FEM simulations and experimental results. The experimental results show a high mass sensitivity of $1356.81 \text{ Hz.cm}^2/\text{ng}$ ($175.42 \mu\text{m}^2/\text{ng}$) and limit of detection of $18.7 \times 10^{-6} \text{ ng}/\mu\text{m}^2$ for a 773 MHz resonator. This high mass

sensitivity arises from the high resonance frequency which can be independently set from the resonator thickness for this special design. The obtained mass sensitivity of the developed sensor in the air is much higher than the state of the art mass sensors and shows the high potential of the developed piezoelectric transducers for mass sensing applications. However, the sensor performance in the liquid environment (Fluorinert) shows a very low Q-factor which eliminates the possibility of using this type of sensor for biosensing (Chapter 4). The performances of developed sensors, the capacitive Lamé-mode sensor and AlN checker-mode resonator are compared in Table 6-1.

Table 6-1 Comparison of the performance of the Lamé-mode and AlN checker-mode resonators based on the obtained results of chapters 3 and 4

	Capacitive Lamé-Mode Resonator	AlN Checker-Mode Resonator
Typical Resonance Frequency	38.7 MHz ($L = 100 \mu\text{m}$)	773 MHz ($\lambda = 12 \mu\text{m}$)
Q-factor in air ($P \cong 1\text{atm}$, Temp = 22°C)	1800	956.8
Q-factor in Liquid	280 (under water droplet)	27 (Fluorinert)
Mass Sensitivity in Air	$87.76 \mu\text{m}^2/\text{ng}$	$175.42 \mu\text{m}^2/\text{ng}$
Mass Sensitivity in Liquid	$79.53 \mu\text{m}^2/\text{ng}$	-
Limit of Detection	$1.36 \times 10^{-6} \text{ ng}/\mu\text{m}^2$	$18.7 \times 10^{-6} \text{ ng}/\mu\text{m}^2$

The mass sensitivities characteristics of the AlN checker-mode are better than the Lamé-mode resonator. However, the Q-factor value of the Lamé-mode resonator under droplet of the water is higher than AlN checker-mode resonator. For this reason, the application of the Lamé-mode resonator as a biological sensor in liquid environment has been investigated in chapter 5. The specific binding of biotin and streptavidin immobilized on the resonator surface is used for the sensor characterization. Different to the common methods that the sensor surfaces are coated by a thin gold layer for the immobilization of

probe molecules, the biotin molecules is directly immobilized on the silicon surface using APTES. A significant frequency change is observed when the interaction occurs on the surface of biotin functionalized resonators as opposed to minor frequency change of unfuctionalized (BSA blocked) sensor. The mass sensitivity of $87.76 \mu\text{m}^2/\text{ng}$ and Q-factor of 280 in aqueous environment are demonstrated. Due to the small size of the resonator they can be mass produced and arrayed for analysis of multiple targets simultaneously. These results encourage the efforts on using Lamé-mode resonator for fast and label free analysis of biomolecular interactions.

6.2 Proposal for Future work

Some possible approaches are proposed to improve the performance of resonant microstructures as mass sensitive sensors in future. Clearly, a creative design of the resonant microstructure, coating strategies, which are optimized for the micro-scale structure, and clever compensation techniques are keys to any successful commercialization of a resonant mass-sensitive biochemical sensor.

First way of improvement the sensor performance is to derive analytical equations for mass sensitivity and Q-factor of the resonator in the liquid environment. Despite of many simplifying assumptions, the derived analytical models in this thesis showed good agreement with FEM simulation data and experimental results, especially in air. However, a more careful and systematic analytical analysis is required to explain the differences between estimated and measured mass sensitivity and Q-factor in air and water. Especially, the mass loading effect by the surrounding fluid and the slide film dampings have to be considered to derive an improved analytical model for the square resonators.

Secondly, a precise estimation for mass sensitivity of the sensor when exposed to the bio entities is required. The potential application of the designed resonators for biological sensing in a liquid environment has been demonstrated experimentally using the specific binding of biotin-streptavidin immobilized on the resonator surface. The resonators exhibited a stable response when exposed to the streptavidin solution and showed an approximately 12 kHz frequency change upon coating of 3.43 ng of streptavidin in a 10^{-7} M solution. However, a precise estimation of the sensor sensitivity and resolution in detecting the antibodies in a solution with known concentration has not been done. This requires an improved repeatability of the sensor response, and in particular a careful design of a flow cell and the measurement setup.

Thirdly, due to the small size of the resonator, they can be mass produced and arrayed for analysis of multiple targets simultaneously. The arrayed type sensor will need a more complicated electronics, but these results encourage the efforts on using arrayed form of Lamé- mode resonators for fast and label free analysis of biomolecular interactions.

Finally, some approaches for improving the mass detection capability of microresonators based on the reducing the environment noises can be considered in new designs. For instance, the resonance frequency drifts caused by temperature changes can be compensated. The compensation method can be based on a controlled stiffness change introduced by an additional feedback loop. By compensating for subtle temperature changes, the resonance frequency variation caused by mass changes, i.e. the effective sensor signal, can be assessed more precisely. Influences of other environmental factors (e.g. humidity) can be also investigated as well.

Appendices:

Appendix A: Detailed Fabrication Steps

Step	Stage	Recipe	Step Description	Wafers to be processed	Equipment	Measured results & Comments
1	Wafers Start		Test Wafers	S1-S4		P/Boron Resistivity: 0.008- 0.02 ohm-cm
			SOI Wafers	S5-S8		Dopant P/Boron Resistivity: 1-20ohm.cm <100>
			Prime wafers			
			Ordered SOI wafers			
2	QDR Clean		QDR Clean	S1-S8	Wet Bench	
3	LPCVD Nitride Deposition		Nitride Thickness: 3 KA	S1,S5-S8	Semco Furnace	
4	LPCVD Oxide		Oxide thickness: 1.5 KA	First S1; S5-S8	STS-CMOS	
5	1st Litho: PR Coating	LA003	PR - 1 micron w/o BARC	S1 First; S2-S8	Track_for_248_ stepper	
6	Exposure	AMIR/ DEVICE	Reticle ID: AMIR-DEV-ELC-1020	S1 First; S2-S8	Nikon aligner	
7	PR Developing			S1 First; S2-S8		
8	Etch Oxide		Etch 1.5 KA oxide in open windows	S1 First; S5-S8	P5000	

9	Etch nitride	BT-2-N EP+OE 15 Sec	Etch 2KA nitride in open windows	S1 First; S5-S8	P5000	
10	PR Strip	Rcp4		S1 First; S5-S8	Mattson	
11	Etch Silicon		Etch depth: 2.5 microns, (Stop on Box)	S1 First; S2-S8	P5000	
12	Etch nitride	BT-2-N EP+OE 15 Sec	Etch ~1KA nitride in open windows stop on Si	S1 First; S5-S8	P5000	-
13	Inspection		SEM (confirm the etch depth)	S1-S8	DR-SEM	
14	LPCVD Si ₃ N ₄ Deposition		Si ₃ N ₄ Thickness: 100A (S1 & S5) 200A (S2 & S6) 400A (S3 & S7) 800A (S4 & S8)	1st batch: S3,S4,S7,S8 2nd batch: S1,S2,S5,S6	SEMCO Furnace	Two thicknesses in batch, put the rest of the wafers on hold
15	Measurement		Si ₃ N ₄ thickness measurement, Spec: 100-800A. Measure the device wafer, 5 points	S3,S4,S7,S8	OptiProbe/Ellipsometer	Inspection
16	LPCVD PolySilicon Deposition		Thickness of Low Stress PolySilicon: 2 microns	S3,S4,S7,S8	SEMCO Furnace	
17	Implantation		1st: p doped, 60 keV/Boron, $2 \times 10^{15} \text{ cm}^{-2}$ 2nd: p doped, 150 keV/Boron, $2 \times 10^{15} \text{ cm}^{-2}$ 3rd: p doped, 180 keV/Boron, $2 \times 10^{15} \text{ cm}^{-2}$, Tilt= 7 degrees; rot=0	S3,S4,S7,S8	Diffusion Chamber	
18	Annealing		950°C, 1 Hours	S3,S4,S7,S8	Furnace	
19	Measurement		PolySilicon thickness measurement, and sheet resistivity 4 points measurement	S3,S4,S7,S8	OptiProbe	Inspection

20	2nd Litho: PR Coating	LA052	PR - 2 micron w/o BARC	S3 First; S4,S7,S8	Track_for_2 48_ stepper	
21	Exposure	Amir/ELC	Reticle ID: AMIR-DEV- ELC-1020 (Top Box)	S3 First; S4,S7,S8	Nikon aligner	
22	PR Developing			S3 First; S4,S7,S8		
23	Oxide Etching		DHF (1:100), 30 Sec (2 times)	S3,S4,S7,S8	Remove native oxide	
24	Deep Reactive Ion Etching	Recipe THM - 5mins $\pm$ 30 Sec	2 microns PolyEtching, (Stop on Silicon nitride)	S3 First; S4,S7,S8	STS-CMOS	
25	Inspection		Confirm the poly etched Stop on Si ₃ N ₄	S3,S4,S7,S8	DRSEM (EDX)	
26	Nitride Etch	BT-2-N EP+OE 10 Sec	Etch Silicon Nitride from the Front Side	S3,S7->40nm S4,S8->80nm	P5000	
27	Inspection		Confirm the nitride etch, Stop on Polysilicon	S3,S4,S7,S8	DRSEM (EDX)	
28	PR Strip	Rcp4		S3,S4,S7,S8	Mattson	
29	Wet Clean		Piranha (10 mins)	S3,S4,S7,S8	Wet Bench	

Transfer: from Non Copper Pod to Copper Pod						
30	Sputtering Ta+Al		500A of Ta+7500A of Al	S3,S4,S7,S8	Endura PVD	
31	Backside Cleaning			S3,S4,S7,S8	Sez Cleaning	
Transfer: from Cu Pod --> Non Cu Pod						
32	3rd Litho: PR Coating	LA003	PR - 1 micron w/o BARC	S3 First; S4,S7,S8	track_for_248_ stepper	
33	Exposure	AMIR/P AD	Reticle ID: AMIR_PAD_LK_3040 GDS#30 (top)	S3 First; S4,S7,S8	Nikon aligner	
34	PR Developing			S3 First; S4,S7,S8		
35	Etch Al/Ta	Al: 7500A (E.P) Ta: 500A (O.E)	Al/Ta etch (Passivation only)	S3 First; S4,S7,S8	Centura	
36	PR Strip		PR strip	S3,S4,S7,S8	Mattson	
37	Wet Clean	CV015		S3,S4,S7,S8	Verteq	
Transfer to Cu Contaminated Clean Room						
38	4th Litho: PR Coating		PR - 10 microns w/o BARC (Back Side)	S3 First; S4,S7,S8	SVG Track	
39	Exposure		Front to Back Alignment Mask ID: AMIR_RCT_CHM Expose on Back Side	S3 First; S4,S7,S8	EVG Aligner	
40	PR Developing			S3 First; S4,S7,S8	SVG Track	

41	Plasma Therm		Opening 2um Poly+(20-80nm+2KA) nitride in the windows Back Side	S3 First; S4,S7,S8		
42	PR Strip		Wet Stripping	S3 First; S4,S7,S8		
43	Anisotrpoic Wet Etching		Etchant: KOH 80°C Etching Holder	S3 First; S4,S7,S8		
44	Inspection		Confirm the Cross Section, chamber opening	S3 First; S4,S7,S8	Wafer cutting, FIB	
45	Plasma Therm		Etch 2um buried oxide and release the resonator	S3 First; S4,S7,S8	Plasmatherm	
46	Dicing			S3 First; S4,S7,S8		

Appendix B: The designed PCB layout in ALTIUM DESIGNER software:

Figure A-1: The designed PCB layout in ALTIUM DESIGNER software

Reference:

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