PARYLENE-COATED PIEZOELECTRICALLY-ACTUATED SILICON DISC RESONATORS FOR LIQUID-PHASE SENSING

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ABSTRACT

In this work a novel surface treatment is applied to thin-film piezoelectric-on-silicon disc resonators that enables liquid phase operation of the resonator while maintaining a high quality factor (*Q*). By coating the backside and the surrounding trenches of the resonator with Parylene-C, a biocompatible insulation layer is formed that completely avoids the penetration of liquid into small gaps while enabling the surface functionalization for various sensing purposes. The effect of viscous damping on different vibration modes of the disc resonator is investigated and a high order contour mode is identified at 17.2 MHz which exhibits the least drop in the quality factor (*Q*) when operated in liquid media. A record *Q* of 440 is measured for this resonance mode in water, corresponding to a ~55% reduction from the *Q* measured in air. Such high *Q* values in water facilitate implementation of high resolution liquid-phase mass sensors for biomedical or industrial applications.

KEYWORDS

MEMS resonator, piezoelectric devices, liquid phase, resonant sensors, high quality factor, real-time sensing.

INTRODUCTION

Microelectromechanical (MEMS) resonant sensors are rapidly finding new applications in diverse fields spanning from medical diagnosis and drug discovery to auto industry and monitoring of industrial processes due to their small footprint, low cost, and high accuracy. These sensors operate based on a transduction from a physical parameter of interest, e.g. change in the mass due to the binding of a targeted substance, to an electrical signal, e.g. resonance frequency. The frequency-based output signal is attractive as it will enable a direct digital readout. This delivers a great advantage compared to the widely employed optical detection schemes [1], since they require relatively expensive and bulky components for detection and readout that may not be easily accessible or may involve a labeling process that introduces complexities and uncertainties. Despite the numerous advantages of MEMS resonant sensors, designing such devices that can operate in liquid media remains challenging due to viscous damping from liquid contact and loss of the signal as a result of undesired feedthroughs and short circuits in the liquid media. Accordingly, MEMS resonators still lag behind their major rival, quartz crystal microbalances (QCM), since they offer much larger quality factors (*Q*) and consequently higher resolution [2]. The superior performance of QCMs can be attributed to vibrating at shear modes which are less impacted by viscous damping compared to the flexural and lateral modes since no force normal to the surface of the resonator is exerted to the molecules in the liquid.

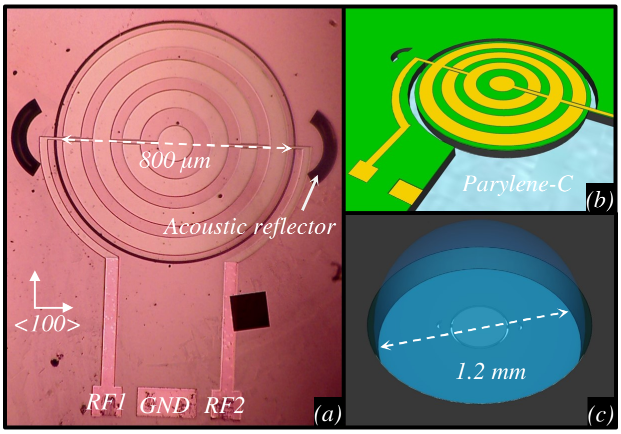


Figure 1: (a) micrograph of the proposed sensor, (b) schematic of the sensor with portions of the substrate removed to expose the Parylene-C layer drawn in light blue, (c) the backside microwell for liquid insertion.

Accordingly, operation of the resonator at a resonance mode with minimum out-of-plane displacement combined with techniques to prevent the liquid from penetration into the surrounding gaps can pave the way toward the application of MEMS resonators in liquid sensing and research in this topic is ongoing with limited success [3-6]. Pursuant to the trend of increasing the *Q* of such sensors, here we propose to investigate viscous damping of multiple resonance modes of a thin-film piezoelectric-on-silicon (TPoS) [7] disc resonator in liquid while a thin layer of Parylene-C is incorporated in the body of the resonator that isolates the device and the surrounding gaps from the liquid. Our investigation reveals the efficiency of this technique in improving the liquid-phase *Q* of the resonator. The measured higher *Qs* improve the reliability of the oscillator circuit built from the resonator and the resolution of the overall sensor.

DESIGN

The transduction from the physical feature of interest to frequency is performed by a TPoS disc resonator, comprising single crystalline silicon (Si) and a thin layer of 20% scandium-doped aluminum nitride (ScAlN) sandwiched between two molybdenum (Mo) electrodes as the main resonant body and the transducer layer, respectively. The top electrode is patterned to allow for simultaneous excitation and sensing while matching the stress profile of multiple resonance modes, as shown in the micrograph picture in Fig. 1. The lateral boundaries of the resonator are defined by narrow air gaps and two thin tethers that support the suspended body. These two tethers are aligned to the [100] silicon plane which results in the minimum distortion of the radial resonance modes [8] and acoustic reflectors are positioned close to them so as to mitigate the tether loss [9, 10].

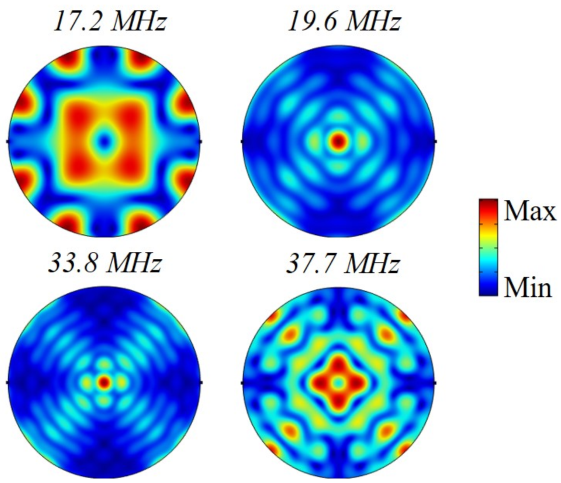


Figure 2: COMSOL FEA of the total displacement profile of the excited modes displaying the tether placement at nodal points.

The simulated displacement profile of the excited modes obtained from finite element analysis (FEA) is shown in Fig. 2, highlighting the placement of the tethers at the nodal points of the resonance modes. The backside of the resonator is a relatively wide cavity that forms a microwell where the liquid under test is injected with a high gauge syringe needle. The backside cavity is isolated from the resonator with a sub-micron layer of Parylene-C, a chemically inert polymer that forms barriers impermeable to gases and liquids as thin as a few tens of nanometers [11]. Incorporating such coating layer not only completely eliminates the penetration of the liquid in the narrow trenches and the subsequent irreversible loss of the acoustic energy, but it also insulates the electrical conduction pathways from the liquid, which aids in lowering the parasitic feedthroughs; these result in improvement of the Q and signal to noise ratio, both of which are crucial for the improved resolution and reliability of the sensor. The Parylene-C coating layer is hydrophobic with a large water contact angle whereas a hydrophilic coating is desired for proper wetting and surface functionalization of the sensor; a short oxygen plasma exposure efficiently breaks the surface bonds of the coating, resulting in hydrophilicity [12] and enabling the application of binding agents on the film [13]. The radii of the disc resonator and the backside microwell are 400 µm and 600 µm, respectively.

FABRICATION PROCESS

The proposed sensors are fabricated in a 5 mask process, which is summarized in Fig. 3. The fabrication starts with sputtering a 20 nm layer of AlN as a seed layer on a silicon-on-insulator wafer, with 40 µm high resistivity Si layer, which significantly improves the c-axis growth of the ScAlN layer in the following step. Next, a stack of Mo/ScAlN/Mo (0.1/1/0.1 µm in thickness) is sputtered on the substrate. The top electrodes are defined by dry etching using SF_6 and masked by photoresist (PR) followed by wet etching the piezoelectric layer – in order to form contacts to the bottom electrode – in a heated TMAH based solution and masked by a plasma enhanced chemical vapor deposited (PECVD) SiO_2 layer. Next, a thin layer (~100 nm) of Cr/Au is deposited using electron beam evaporation and lift off to improve the electrical contacts to the top and bottom electrodes. In order to define the boundaries of the device, the stack of ScAlN and Si are etched with a chlorine based RIE/ICP and DRIE, respectively which is masked by PR and a PECVD SiO_2 layer. The backside of the wafer is masked with a PECVD SiO_2 layer and etched with DRIE, forming a wide cavity where the liquid droplet is inserted and exposing the 1 µm buried oxide (BOX) layer. Using chemical vapor deposition at room temperature, a ~500 nm sheet of Parylene-C is conformally deposited on the backside of the wafer. Finally, the wafer is immersed in buffered oxide etchant (BOE) for a certain time so that the penetration of BOE from the top side trenches release the device from the BOX layer and leave the device anchored to the substrate with two tethers and the Parylene-C sheet. The Parylene-C coating layer is treated with O_2 plasma at 150 W for 1 min to promote the hydrophilicity of its surface.

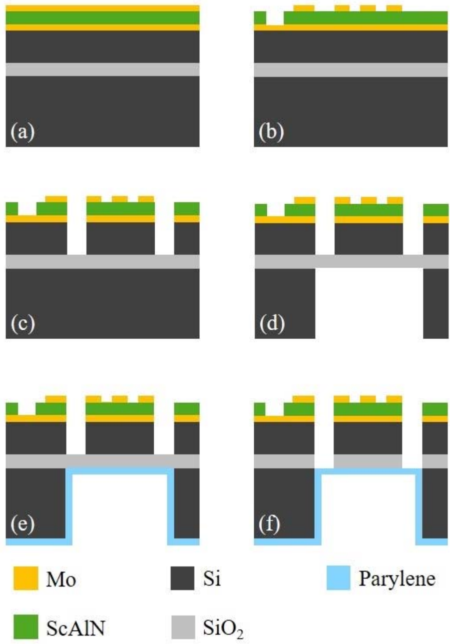
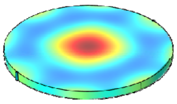
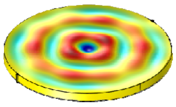
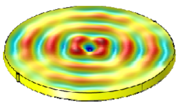
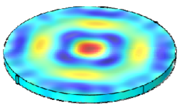


Figure 3: Fabrication process flow of the proposed sensors: (a) the whole stack (b) pattering the top electrodes and contacting the bottom electrode (c) dry etching the device stack (d) etching the backside of the wafer (e) depositing the Parylene-C coating layer (f) releasing the device in BOE.

EXPERIMENTAL RESULTS

In order to characterize the performance of the sensor, it is flipped and mounted on a customized substrate with an opening aligned to the backside microwell to facilitate the insertion of liquid droplets. The electrical contact pads of the resonator are wire bonded to the substrate and the substrate is connected to the ports of a Rhode & Schwarz ZNB 8 network analyzer via coaxial cables. The resonator is excited into vibration via one port while the second port transduces the vibrations back to voltage and the frequency response (i.e. S_{21}) is measured.

Table 1: Performance of the excited modes while coated with Parylene-C in air, water, and after removing the coating.

	Frequency (MHz)	***Resonance Mode***	***Stress Profile FEA***	***Q without Parylene***	***% decrease***	***Q with Parylene in air***	***% decrease***	***Q with Parylene in water†***
I	17.2	4-1 Contour		1540	36	980	55	440
II	19.6	Flexural		1990	49	1020	×	×
III	33.8	Flexural		2570	68	810	×	×
IV	37.7	0-2 Contour		2330	59	945	91	85

† Time averaged once the microwell is covered and the frequency response is stabilized

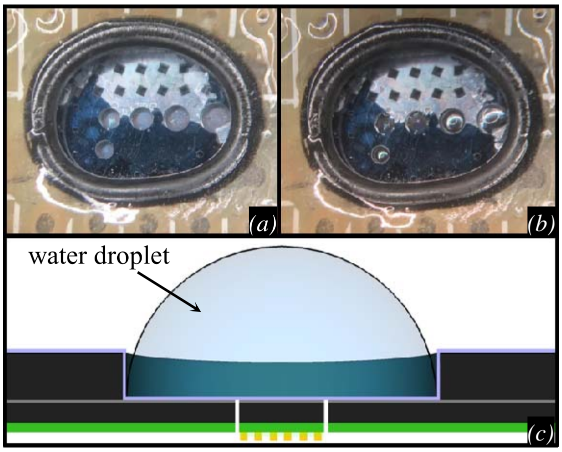


Figure 4: Optical image of the back side of the resonator (a) before and (b) after being filled with DI water. Cross-sectional schematic of the device showing a water droplet in the back side microwell is shown in (c).

Prior to filling the microwell with water, four resonance modes are efficiently excited in the range of 15 MHz to 40 MHz, two of which are flexural modes while the other two are radial contours. The corresponding resonance frequency, averaged *Q*, and stress profile obtained from COMSOL simulations are presented in Table 1. Next, the backside microwell is filled with deionized (DI) water using a 34G syringe needle as depicted in Fig. 4. Upon filling the microwell, large variations in the frequency response are noticeable which stem from the evaporation of water [14]. This problem is addressed by placing a small silicon cap to fully cover the microwell and by doing so, the saturation vapor pressure is reached promptly, resulting in a stabilized and reliable measurement. The results obtained from the device with the highest averaged *Q* in water after the stabilization of the frequency response are presented in Table 1 for comparison with the results from the device in air.

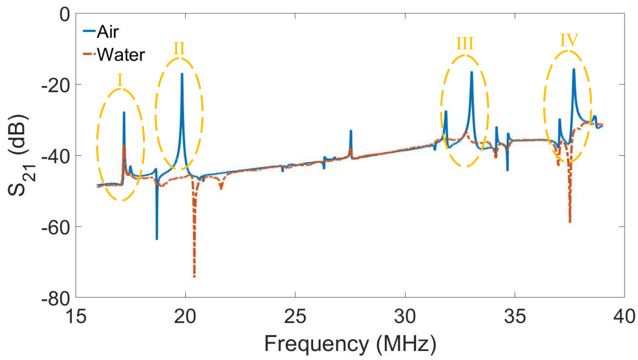


Figure 5: Wide-span frequency response of the resonator, comparing the amplitude of different modes in the air and once the microwells are filled with water.

The frequency response of the resonator across the total span of study before and after adding DI water is plotted in Fig. 5. In accordance with the expectations, viscous damping due to the insertion of water into the microwell drastically dampens the flexural modes since they have major out of plane displacement components. However, the radial contour modes, especially the 4-1 mode at 17.2 MHz, are less susceptible to viscous damping due to their negligible out of plane movements. Fig. 6 shows the frequency response (magnitude of the S_{21}) of the resonator with the highest *Q* in the 4-1 radial contour mode in air and once the microwell is filled with DI water. For this resonator and vibration mode the *Q* is decreased from 980 to 440 and the loss is increased by ~6 dB, resulting in an increase in the motional resistance from 3.4 kΩ to 6.5 kΩ. Once the performance of the Parylene-C coated device is characterized, the coating layer is etched away in RIE using O_2 plasma. By doing so, the initial influence of adding the coating layer on the performance of the excited modes of vibration is achieved. The averaged *Q*s measured after the removal of the coating layer are presented in Table 1 for comparison, as well.

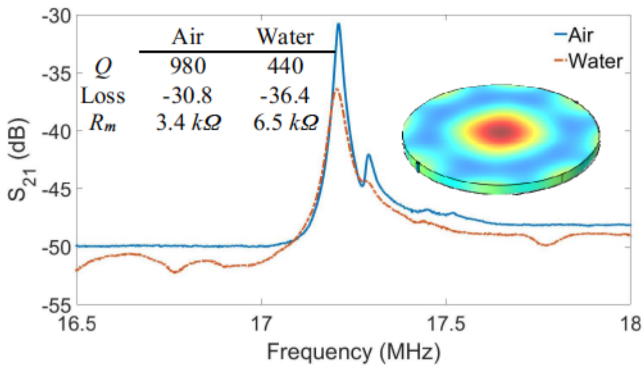


Figure 6: Frequency response of the 4-1 radial contour mode with the highest Q in water and compared with air, showing a strong resonance peak in the water; inset shows the stress profile simulated by COMSOL.

The results exhibit that the 4-1 radial contour mode is least affected (55% reduction in the Q) by the addition of the coating layer which is in agreement with the fact that this resonance mode is more immune to damping from being in contact with a liquid medium or some other layer of an elastic material.

CONCLUSIONS

TPoS disc resonators having a wide backside cavity that is coated with a thin layer of Parylene-C from the backside were investigated as a possible alternative for QCMs. Such devices could potentially have various applications in liquid-phase sensing by maintaining high Qs while benefiting from the advantages of batch microfabrication. The impermeable coating layer of Parylene-C does not significantly degrade the performance of the resonant sensor at the optimal vibration mode, however, it completely suppresses the liquid from entering the narrow gaps surrounding the resonator and from adding to the parasitics associated with liquid coverage over the electrical contacts. This results in improvement of Q, achieving a record Q of 440, and a low motional resistance of 6.5 kΩ. Such performance is crucial for embedding the sensor within an oscillator circuit for enhanced resolution that is intended for biomedical and industrial mass and real-time viscosity sensing applications, once the sensors are functionalized with the proper binding agents.

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