

Layered SiO_2 / LiNbO_3 / Si SH-SAW Biological Sensor for Operation in Water

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Summary — Surface Acoustic Wave (SAW) sensors have raised an interest for biological sensing applications. Operation in water requires exploiting Shear Horizontal SAW (SH-SAW), which however suffer from losses due to radiation in the substrate. Therefore, we adopt here a resonator structure consisting in a LiNbO_3 layer bonded to a Si substrate and covered by a thin SiO_2 layer to provide electrical insulation when in contact with water. The resonator operates at 378 MHz and its quality factor was evaluated in air/water operation with a Q_{water} of 280.

Keywords—Shear Horizontal SAW (SH-SAW); biological sensor; LiNbO_3

I. INTRODUCTION

The monitoring of water quality is a public-health issue. It requires cheap and robust sensors capable of detecting the presence of biological pathogens in liquid samples. Acoustic sensors are one of the possible technologies that can address these needs. However, the development of an acoustic wave device for biological detection poses several challenges, notably, the operation in water-based complex media that reduces the performance of such devices due to acoustic wave radiation in the fluid and viscous losses [1].

Quartz Crystal Microbalance (QCM) based biosensors have been used for decades now. Yet its usage is limited because of its low mass sensitivity due to large size low frequency of operation [2]. Some studies have been carried out on QCMs operating at higher frequencies, such as 56 MHz and 170 MHz to increase mass sensitivity (S_m) [3, 4]. However, the drawback is that when bulk waves are used, an increase in S_m is accompanied by a decrease in the active area, thus in number of biological samples that can attach to the sensor.

To overcome these limitations, Surface Acoustic Wave (SAW) devices have been proposed. They concentrate the acoustic energy close to the surface, increasing the mass sensitivity, while having an active area spanning over multiple wavelength. SAW sensors usually rely on Rayleigh or Shear Horizontal (SH) propagation modes [3]. The latter is better suited for biological sensing applications because its displacement field does not couple to bulk waves in water. Thus, it can be used with minimal water radiation losses. Its drawback is, however, a larger penetration depth in the bulk substrate compared to Rayleigh waves, hence a comparatively lower sensitivity, and relatively large propagation losses due to coupling to bulk waves in the piezoelectric substrate [6]. Love waves solve these issues by confining the waves in a guiding

layer inserted in between the piezoelectric substrate and the fluid. SiO_2 is the usual material of choice for such a layer. However, minimizing radiation losses and maximizing sensitivity requires relatively large thicknesses (in the order of half to one wavelength), which causes integration issues related to the large thickness to etch to reach contacts to electrodes or to residual stress in the layers [7].

To overcome these limitations, the present work focuses on a layered SH-SAW sensor based on a LiNbO_3 / Si substrate, with AlSi electrodes covered by a thin SiO_2 layer. The SiO_2 layer acts here as an electrical insulator and a surface on which to graft bioreceptors.

II. METHODS/RESULTS

The combination of a high-acoustic velocity substrate with a single crystal piezoelectric layer leads to a confinement of the acoustic waves in the piezoelectric medium, reducing radiation to the substrate [8]. Fig. 1 shows the root mean square (RMS) displacement profile versus depth of the resonator obtained from finite element simulations. The layered configuration consists in a SiO_2 ($h=0.01\lambda$) / $Y+36^\circ$ -cut LiNbO_3 ($h=0.7\lambda$) / SiO_2 ($h=0.1\lambda$) / Si structure with Al electrodes ($h=0.01\lambda$) and it was compared with a similar structure based on a bulk LiNbO_3 substrate. The penetration depth of the SH SAW is reduced to 2λ in the layered configuration whereas in the bulk configuration it is greater than 3λ . The evanescence in the Si substrate indicates a better confinement of the acoustic energy close to the surface.

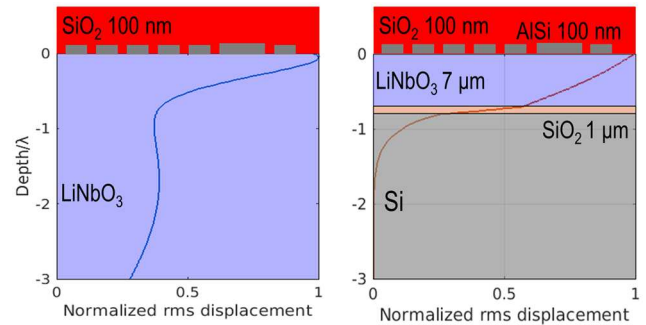


Fig. 1 Calculated normalized displacement versus depth. Top SiO_2 and AlSi (electrodes) layers are out of scale for better visualization.

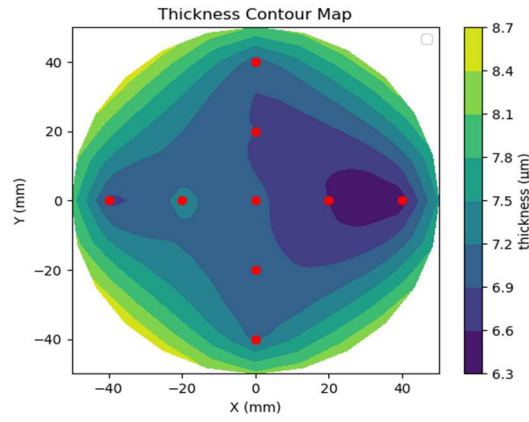


Fig. 2 Ellipsometry map of the LiNbO₃ thickness. Red dots indicate the measurement points and a radial basis function was used to interpolate the data.

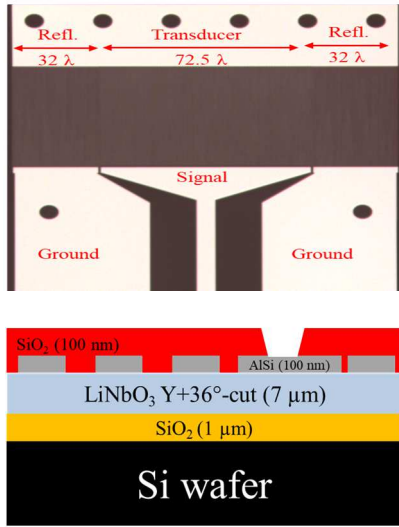


Fig. 3. Optical microscope image and schematic cross-sectional view of the SH-SAW sensor.

The fabrication for the SH-SAW resonators consists in a two masks process. Initially, a 100 mm Y+36°-cut LiNbO₃ wafer is directly bonded onto an oxidized Si substrate (1 μm-thick SiO₂). It is then mechanically thinned by grinding and chemical mechanical polishing (CMP). Fig. 2 shows the LiNbO₃ thickness distribution measured by reflectometry. Although the target thickness was close to 4 μm, we stopped at a thickness of 7 μm because of the difficulty to accurately control the end thickness of the piezoelectric film due to the non-uniformity of the thickness layer throughout the wafer. A 100 nm thick AlSi layer is deposited and 5 μm pitch electrodes are patterned using UV photolithography and wet etching. A SiO₂ protective film is deposited by Plasma Enhanced Chemical Vapor Deposition (PECVD) at 300 °C and patterned by UV photolithography and reactive ion etching to open electrical contacts to the electrodes. Fig.3 shows the final stack of the device and a microscopic view of one patterned

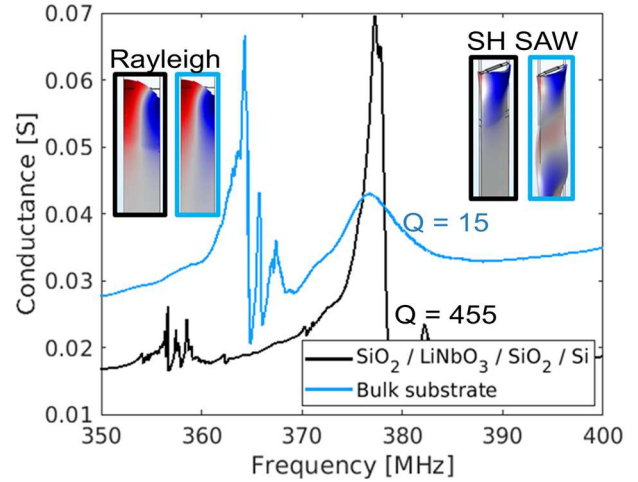


Fig. 4 Conductance of measured SAW resonators implemented on bulk LiNbO₃ Y+41° (blue) and on SiO₂ (h=0.01λ) / Y+36°-cut LiNbO₃ (h=0.7λ) / SiO₂ (h=0.1λ) (black), propagation Z-17° in both cases, operating in air. A 10 μm wavelength is considered. Inset, the modal displacements for the layered (black outline) and bulk resonators (blue outline).

resonator. In parallel, SH-SAW resonators based on bulk substrates have been processed similarly to provide a reference.

After fabrication, the resonators have been measured using GSG coplanar probes connected to a vector network analyzer. Fig. 4 shows the corresponding electric response. In the frequency range between 350-400 MHz one can observe two resonances. The first, at 357 MHz, corresponds to the Rayleigh wave mode. The second, at 379 MHz, corresponds to the SH-SAW mode. The quality factor (Q_{air}) of the SH-SAW is calculated from the full width at half maximum of the resistance peak as 455 and 15 for respectively the devices based on layered LiNbO₃/SiO₂/Si and the bulk LiNbO₃ substrates. This confirms the relatively large propagation losses for the SH-SAW on a bulk substrate, and their reduction on the layered substrate.

Fig. 5 compares the conductance measured for the layered resonator operating in air and in water after depositing a drop

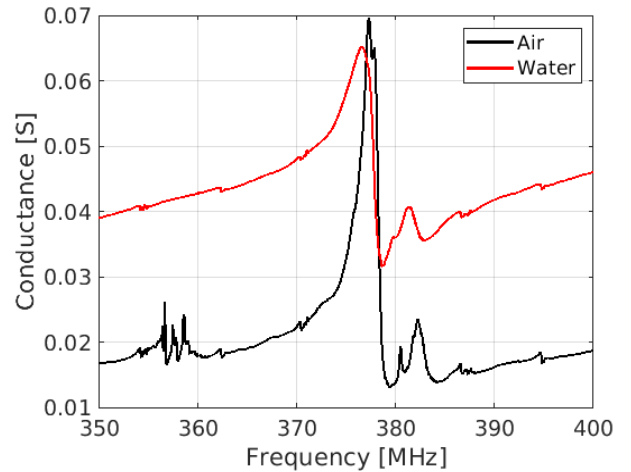


Fig. 5 Experimental electric response in air (black) and water (blue). For resonator with SiO₂ (h=0.01λ) / LiNbO₃ Y+36°(h=0.7λ) / SiO₂ (h=0.1λ), propagation Z-17°.

onto the SAW resonator. After addition of water, the Rayleigh mode nearly disappears, while the shear horizontal mode is only damped by viscosity, with a quality factor reduced to 279.

For biological sensors, the mass sensitivity is a critical parameter that quantifies the relative frequency shift induced by a change in mass in the system. By incorporating a thin layer of mass per unit surface of 10^{-5} kg/m² and performing an eigenfrequency analysis using a 2D periodic finite element model of the resonator, we calculate the mass sensitivity as:

$$Sm = \frac{\Delta f}{\Delta m} \times A$$

where Δf and Δm are respectively the change in frequency and the added mass and A is the active area where mass is deposited. Table 1 summarizes the sensitivities obtained respectively for the layered and bulk SH-SAW.

Table 1 Calculated mass sensitivities for layered and bulk SH-SAW devices

Layout	Sm [Hz×cm ² /g]
Layered	9×10^{11}
Bulk	5×10^9

III. DISCUSSION/INTERPRETATION

As expected, the SH mode, as opposed to the Rayleigh mode, is not suppressed when operating in water. Nonetheless, viscous losses are introduced when the system is contact with water as evidenced by the reduction in Q from air to water. Operation in water also increases the static capacitance from 19.5 to 32.4 pF, which can be explained by the higher dielectric constant of water. This effect may be attenuated by increasing the thickness of the top SiO₂ layer.

Concerning the mass sensitivity of each device, the two SH SAW operate at almost the same frequencies. As a result, the increase in sensitivity is mainly due to the improved confinement of the acoustic energy near the surface, which leads to a reduction in the effective mass of the resonant system.

Table 1 displays the theoretical mass sensitivities for the layered and bulk SH SAW resonators. The layered resonator is expected to be more sensitive, with a 100-fold increase in mass sensitivity. However, it is important to note that the periodic resonator model provides an ideal value for the mass sensitivity ignoring characteristics of the resonator device, such as number of IDT, reflectors and aperture length, which could affect differently the sensitivities of the two modes.

IV. CONCLUSIONS

We have fabricated layered SH-SAW devices based on SiO₂/LiNbO₃/SiO₂/Si, compatible with operation in liquid for biosensing applications and benefiting from reduced bulk wave radiation in the substrate compared to similar devices based on bulk piezoelectric substrates. The better confinement of the SH wave close to the surface not only increased the quality factor from 15 to 455 but is also expected to increase the mass sensitivity by a factor of 100. A proper optimization of the

resonators layouts and a more careful control of the piezoelectric layer thickness would help improving quality factors beyond those measured here (455 in air, 279 in water).

Future work will also focus on the surface functionalization and integration of these sensors into a microfluidic system to perform biological analysis and ultimately perform real-time bacteria detection on water samples.

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