Love Wave Fully Integrated Lab-on-chip Platform for Food Pathogen Detection - LOVE-FOOD

(Contract No 317742 – Starting Date: 1 September 2012)

Deliverable D2.3

Biochips fabrications based on most sensitive SAW devices

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DELIVERABLE SUMMARY SHEET

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Date	Version	Description
27/02/14	1.1	Initial draft: lithium tantalate devices
07/03/14	1.2	Addition of quartz device measurements
14/03/2014	1.3	Diced devices + 310 MHz quartz device
17/03/14	1.4	Coordinator approves and submits to EC officer

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Executive Summary

The work described in the current document is related to WP2, Task2.3. It deals with the fabrication of biochips which will be based on the most sensitive SAW devices described in D2.2. While a clear decision regarding the use of SAW delay line devices instead of resonators was taken and described in D2.2, the operating frequency has not been finalized yet. Of the 5 frequencies tested and described in D2.2, two were the most sensitive ones. Since further testing was considered necessary in order to come down to the most suitable device for the LOVE-FOOD application, both the 155 and 310 MHz devices were considered for biochip application. In addition, quartz as well as lithium tantalite devices were tested in order to optimize the final platform for liquid based operation. The latter is related to the fact that lithium tantalite devices exhibit significantly lower losses when in contact with liquid and, thus, may be an alternative for the final LOC platform if the quartz biochip does not exhibit the necessary sensitivity.

Main Text

Introduction

After testing various sensor geometries (resonator, delay line) based on a given sensing area and pad layout geometry, time has come to select the final layout of the chips used as sensors as part of the LOVE-FOOD project. Having identified the requirements of the two detection paths (bacteria detection on the one hand, triplicate measurements of DNA PCR output on the other hand), a four-acoustic channel system is selected and the layout is adapted for improved manufacturing capability. Due to clean-room manufacturing facility upgrade at the FEMTO-ST institute (http://www.femto-st.fr/fr/L-institut/Actualite/?eid=238&y=2013, in French), the mask and device manufacturing was delayed by a couple of months at the end of the 2013: although the target manufacturing date was met, no gravimetric sensitivity assessment could be performed within the allocated time due to these delays.

i. Quartz devices

The issue of yield is a core aspect of manufacturing cost of the quartz devices. While the original design provided plenty of space for tuning multiple parameters and was even compatible with a resonator design while keeping a constant footprint needed for the radio-frequency test setup, its excessive dimensions yielded only few devices on a 4-inch wafer as classically processed in the clean room. The final SAW device we have designed provides constant sensing area dimensions (in order to remain compatible with the flow cell design envisioned from the beginning of the project) while shrinking all dimensions that could be reduced. A 4-acoustic channel design was agreed upon and 21 4-channel devices now fit on a 4-inch wafer (Fig. 1). While this is still a small number with respect to filter and resonator designs classically used for radio frequency signal processing, the dimensions of the chip are defined by the sensing area dimension and the low operating frequency.

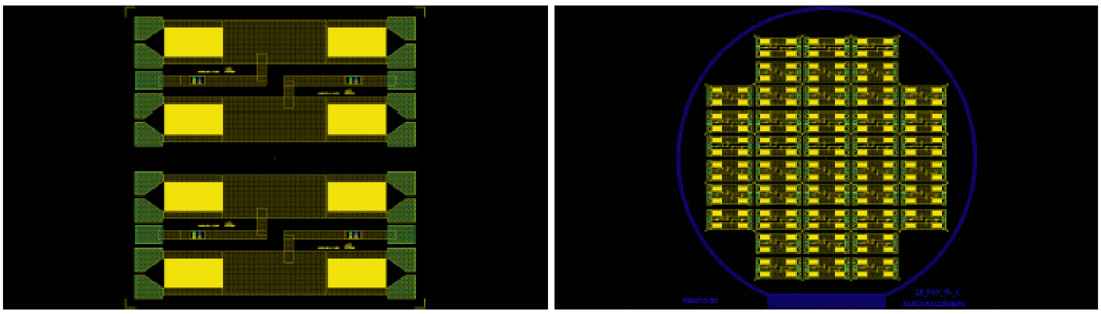
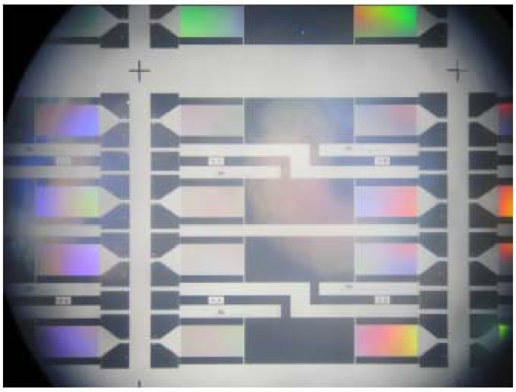
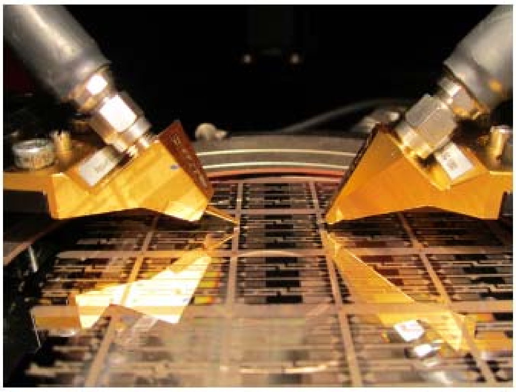


Figure 1: Left: 4-acoustic channel chip. Right: layout of the chips on a 4-inch wafer.

A wafer including sensors operating at both 155 and 310 MHz nominal frequencies has been fabricated following these design considerations, each sensor chip providing four separate acoustic channels as seen in Fig 2 (a). The wafer-scale spin coated 800 nm-thick photoresist layer acting as guiding layer for the acoustic Love mode is only suitable for the 155 MHz devices, which are the only ones characterized at this processing step. Further use of the 310 MHz device will require stripping the current resist layer and individually spin coating a new layer with the appropriate (~500 nm) thickness. Five sensors operating in the 155 MHz range have been characterized and exhibit excellent responses (Fig. 3), with insertion losses in the -20 dB region. Loading the pads with gold provides the necessary wear resistance which was missing in the previous designs. Each channel exhibits similar phase v.s frequency responses as shown in Fig. 4, representative of the behavior of each chip.

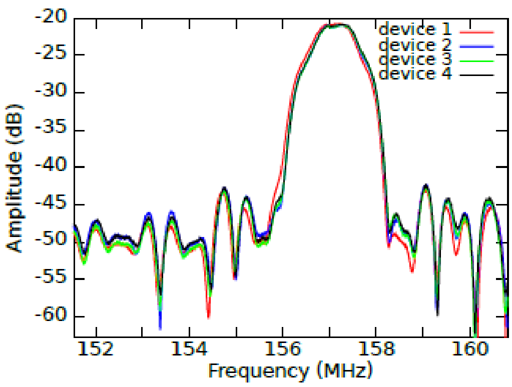


(a)

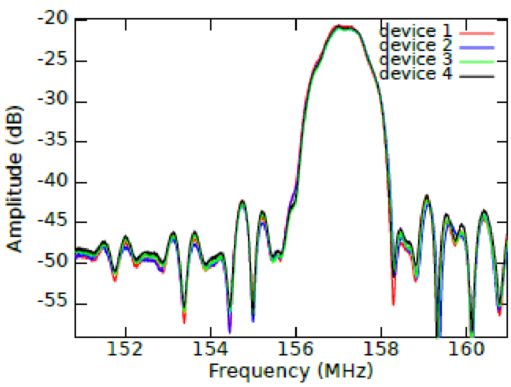


(b)

Figure 2: (a) Each sensor consists of four individual acoustic channels (b) Wafer under test using RF probes connected to a network analyzer.



(a)



(b)

Figure 3: Magnitude spectra of the transfer function of the four channels for two (a and b) sensor chips.

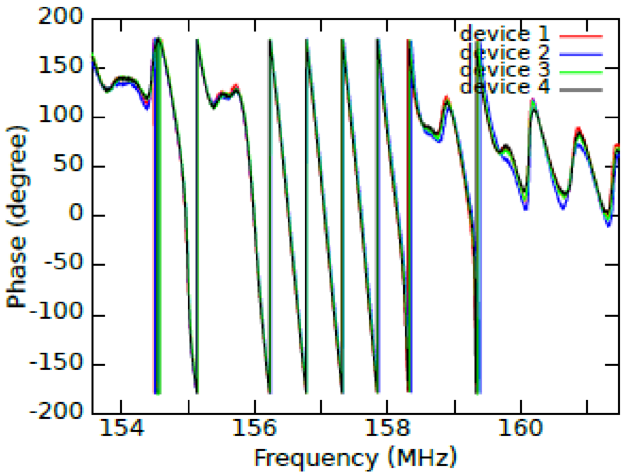


Figure 4: Phase response as a function of frequency.

ii. Quartz diced devices operating at 155 MHz

The acoustic response is maintained after dicing (Fig. 5) although ripples due to the reflections of the acoustic wave on the sides of the chip are now visible. These ripples are eliminated by adding acoustic absorbers after the interdigitated transducers, next to the borders of the chip. The acoustic losses remain excellent and well within the specifications for a stable measurement using either a network analyzer or the embedded electronics supplied by SENSeOR. Five 4-acoustic channels chips operating around 155 MHz were delivered, and an additional set of 310 MHz devices were sent but not characterized since the guiding layer thickness is excessive for guiding such short wavelength waves.

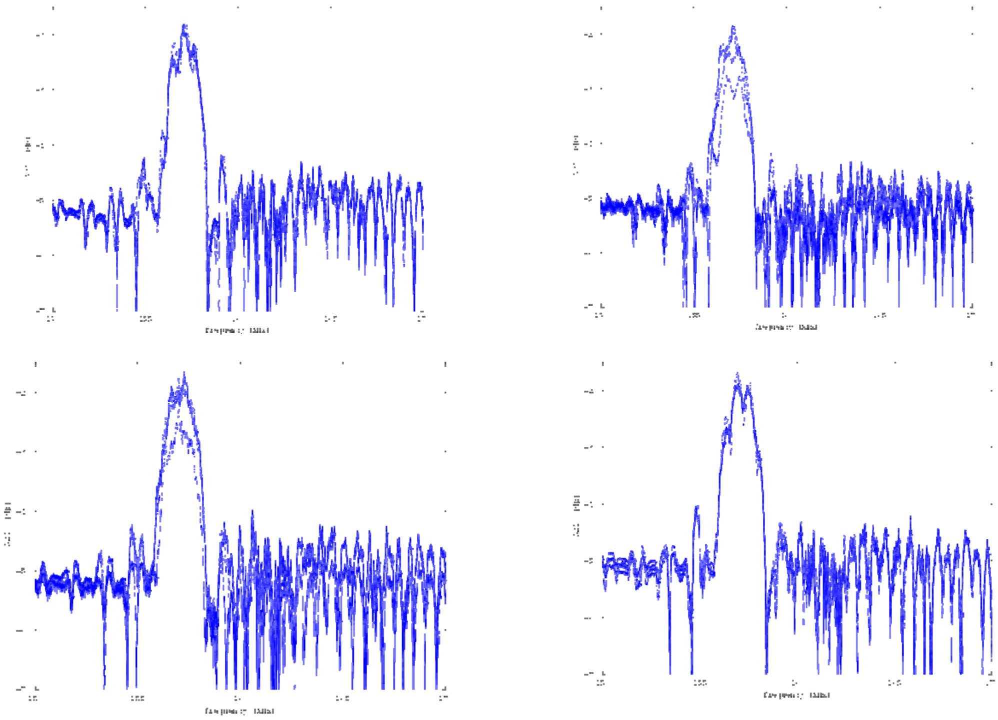


Figure 5: Magnitude spectra of the transfer function of the 4 channels for the 5 diced chips.

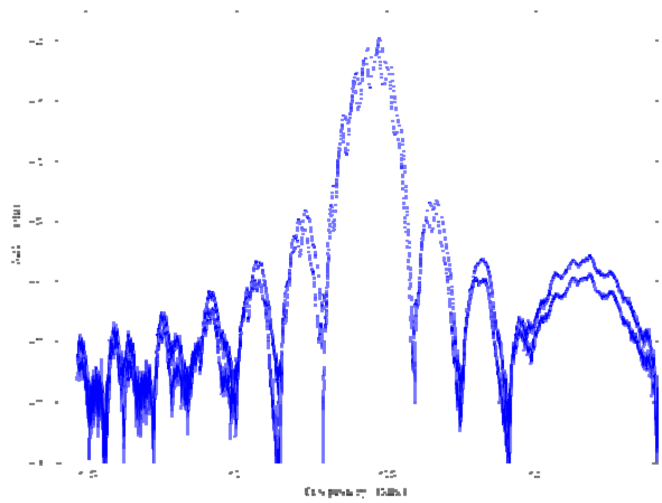


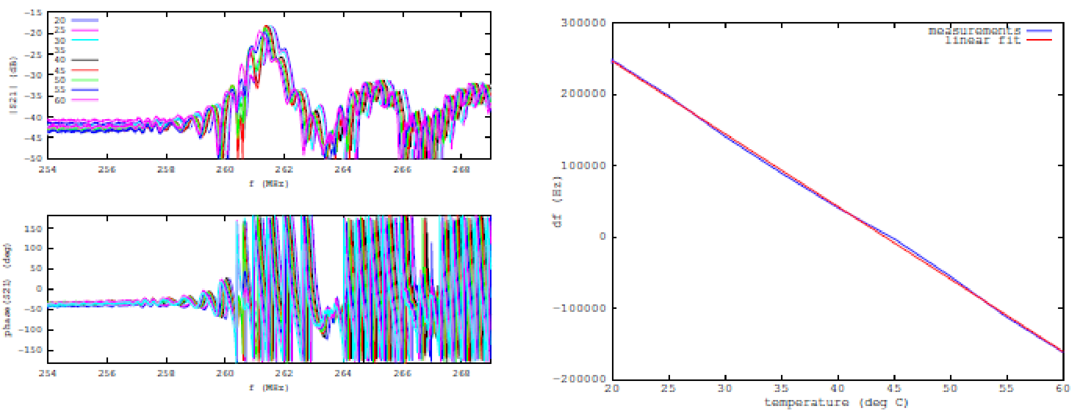
Figure 6: magnitude measurements of the 4-channels of a 310 MHz device coated with 500 nm S1805 photoresist acting as guiding layer.

iii. <u>Quartz devices operating at 310 MHz</u>

One wafer was coated with 500 nm of S1805 photoresist to assess the proper operation of the 310-Mhz 4-acoustic channel devices: the magnitude of the spectra is exhibited in Fig. 6 and is well within the operational range of the embedded electronics.

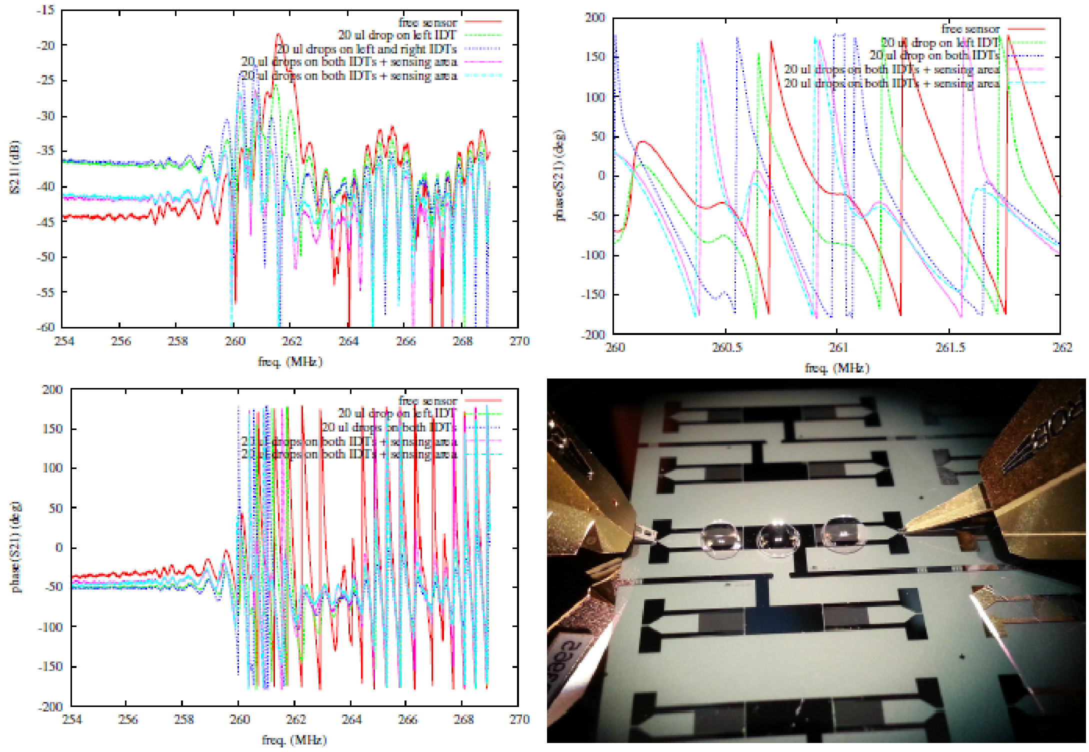
iv. <u>Lithium tantalate devices</u>

Considering the challenging fluidic handling issue when using quartz as a piezoelectric substrate, we have been considering lithium tantalate as a potential candidate for sensing in liquid phase [1, 2]. Indeed, the challenge of fluidic handling on quartz is related to its low relative permittivity: when the interdigitated transducers are covered with water (high relative permittivity of 80), most of the electrical energy will be concentrated in the high-value capacitance provided by water rather than in the low-value capacitance provided by quartz. Water not being piezoelectric, the acoustic wave generation efficiency is reduced and insertion loss dramatically increases, hence the need to restrict water over the sensing area between the interdigitated electrodes. On the opposite, since lithium tantalate exhibits a high relative permittivity of the order of 45, the effect of water coating the interdigitated transducers is much reduced. We wished to experimentally assess whether the acoustic response is still usable under such conditions. The SAW devices were manufactured on YXl/36 lithium tantalate, with a sensing area coated with a metallic lm to convert the pseudo-shear wave to a pure shear wave. A wavelength of λ = 16 µm requires a lithography resolution of 2 µm for a split-finger interdigitated electrode architecture. With a shear wave acoustic velocity of about v ~4200 m/s, the expected operating frequency is in the v/λ = 262.5 MHz range. The 475 nm thick photoresist guiding layer is slightly below the modeled thickness for optimum gravimetric sensitivity, yet provides low acoustic losses, and is nevertheless consistent with the design considerations of [1] in which 500 nm polymer layers are used as guiding layers for a 40 µm wavelength device. The transfer function of the fabricated devices matches expectations (Fig. 7). As predicted by the high temperature sensitivity of lithium tantalate, the guiding layer thermal properties is unable to stabilize the velocity variation with temperature and the resulting sensitivity is in the -37±2 ppm/K range.



<u>Figure 7</u>: Left: magnitude (top) and phase (bottom) of the lithium tantalate SAW device measured at temperatures ranging from 20 to 60 ^{o}C. Right: linear fit of the temperature dependence of the frequency as a function of temperature.

The effect of water coating the interdigitated transducers and the sensing area is assessed in Fig. 8. During this experiment, 20 µl droplets were located over each interdigitated transducer and the sensing area in order to measure the impact on the acoustic losses and phase v.s frequency curve shape. While the acoustic losses remain acceptable under all conditions, the phase curve exhibits additional ripples when 3 droplets cover separately the transducer and sensing area. The ripples are attributed to interferences between the direct wave and the reflections on each air-water interface at the droplet borders.



<u>Figure 8</u>: Left: magnitude (top) and phase (bottom) of the lithium tantalate as the device is coated with increasing number of 20 µl droplets over the interdigitated transducers and the sensing area. Right: zoom on the phase dependence with frequency, emphasizing (bold lines) the usability of the device even when fully covered with water. Bottom-right: experimental setup in which three 20 µl drops are located on the various parts of the SAW device.

<u>References</u>

[1] T. Newman, F. Josse, A. Mensah-Brown, & F. Bender, Analysis of the Detection of Organophosphate Pesticides in Aqueous Solutions Using Polymer-Coated SH-SAW Sensor Arrays, Proc. IEEE International Frequency Control Symposium (IFCS), pp. 620{623 (2013)

[2] F. Bender, F. Josse, R.E. Mohler & A.J. Ricco, Design of SH-Surface Acoustic Wave Sensors for Detection of ppb Concentrations of BTEX in Water, Proc. IEEE International Frequency Control Symposium (IFCS), pp. 628{631 (2013)

Conclusions

Four-acoustic channel quartz-based SAW sensor chips were manufactured following design considerations aimed at optimizing manufacturing yield. All chips exhibit acoustic losses of about 20 dB and a rejection of over -50 dB, well within the expected specification for stable and reliable biosensing measurements. Beyond the quartz-based sensors selected for the LOVE-FOOD project, similar devices made of lithium tantalate - selected for its high dielectric permittivity yet propagating a pure shear wave under appropriate boundary conditions - were manufactured and provided responses in close agreement with the modeled response. While D2.3 met the target manufacturing date of M18, it should be noted that due to clean-room manufacturing facility upgrade at the FEMTO-ST Institute no gravimetric sensitivity assessment could be performed within the allocated time; for this reason D2.4 and D2.5 will be delayed for maximum two months, i.e. will be delivered at M20 and M26 respectively.