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MANUFACTURE OF LOW COST SAW DEVICES FOR CHEMICAL SENSING

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Abstract. *This paper presents the fabrication of a low cost SH-SAW sensor used for chemical sensing. The structure is based on a standard delay line configuration, using a 36^0 rotated Y-cut X-propagating $LiTaO_3$ regular substrate with both piezoelectric and pyroelectric effects. The packaging process and device characterization provided by analyzing the transmission parameters (amplitude and phase) of the surface acoustic wave are also discussed. In the end, there is presented the characterization method of the SAW chemical sensor using the vector network analyzer.*

Keywords: SAW devices, Chemical Sensors, $LiTaO_3$ (36^0YX), piezoelectric substrate

1. Introduction

Acoustic wave devices and technology have received increasing attention during the last years, due to the growing demands for high quality components in the telecommunication field and environmental sensing. Acoustic structures are defined by the wave's propagation mode through/on a piezoelectric crystal. In SAW devices wave propagates (guided/unguided) along the surface of the piezoelectric material. Therefore, these structures are highly sensitive to surface perturbation and can operate in both gas and liquid media. Particle's displacement directions relative to the wave propagation direction depend on the substrate properties. Experimentally, it has been shown that for operation into the liquid media particle's displacement directions normal to the surface of the device suffer from severe attenuation and high insertion losses. Instead, the acoustic waves shear horizontally polarized are more suitable to operate in aqueous environment [1-5]. Specifically, (bio)chemical sensors based on acoustic devices provide excellent sensitivity and good stability.

Chemical sensors for the determination of concentrations of harmful substances in gases and liquids operate, in most cases, by analyzing the transmission parameters (amplitude and phase) of a surface acoustic wave (SAW) that crosses the device.

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Two interdigital transducers (IDTs) are located on both sides of the sensing area, at certain distances from its edges (Fig. 1).

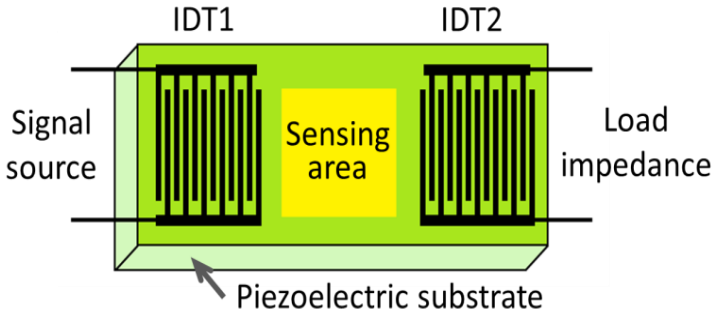

Fig. 1. Structure of a transmission type SAW sensor.

A material with controlled adsorbing properties for the specific analyte is deposited over the sensing area.

The acoustic wave launched by one IDT travels through the sensing area, hence the parameters of the acoustic wave at the terminals of the second IDT are modified under the influence of the actual transmission conditions.

The analysis of these parameters provides data on the magnitude of the parameters that have influenced the propagation.

2. SAW chemical sensor development

The attenuation of the acoustic wave is highly dependent on piezoelectric material. The attenuation increases especially in the case of propagation over long distances relative to the acoustic wavelength. It is therefore essential that the intrinsic loss over the piezoelectric material is as small as possible [6].

The best performing materials from this point of view are quartz (SiO_2), lithium tantalate ($LiTaO_3$), lithium niobate ($LiNaO_3$), zinc oxide (ZnO) and aluminium nitride (AlN) [7]. $LiTaO_3$ is the most suitable for operating in liquid media, especially due to its dielectric permittivity (ε_r), which is closer to that of water.

The wave's propagation mode through this material is shear-horizontal (SH), with 4112 m/s typical velocity. $LiTaO_3$ wafers can be produced with various specifications, such as "black" wafers, with free of pyroelectric discharge, or higher physical strength to withstand processing during manufacture.

The high prices of these wafers result in increased costs of the final product. Therefore, our work focuses mainly on manufacturing of SAW chemical sensors using regular 36°YX $LiTaO_3$ substrate wafers, with both piezoelectric and pyroelectric properties, (whose cost is relatively lower compared to black "wafers), using compatible IC processes as well.

2.1 Design technique

The SAW structure is based on a delay line configuration. Both IDTs have 50 finger pairs, ordered in single-finger configuration with 8.5 μm electrode and gap width. The sensing aria placed between IDTs is 9 mm^2. The device operating frequency is 120 MHz for $LiTaO_3$ 36°Y–cut substrate orientation [8]. Three photolithographic masks were designed using CleWin software for device fabrication. The first mask is used to define the metallic interdigital transducers. The aim of the second mask is to configure the sensing area placed between the IDTs. At the end of the fabrication flow, the third mask is used to give the access to the device pads.

2.2 Technological considerations

The fabrication flow started with the wafer's cleaning process. The first stage consisted in the immersion of the $LiTaO_3$ substrate in piranha solution (mixture of H_2SO_4 and H_2O_2), for removing the organic residues. This process has been achieved during 20 minutes at lower temperature, i.e. 40°C instead of 110ºC, as required by the pyroelectric effect of the substrate. An oxygen plasma cleaning was afterwards performed for surface activation. Using the first photolithographic mask the substrate was prepared for the lift-off process. Using an Electron Beam Evaporation system (Temescal FC-2000), the metallic Cr/Au thin films have been deposited. The 10 nm thickness thin film of chromium has been used as adhesion layer, while the 100 nm thickness gold film was chosen as structural material due to its excellent antibodies immobilization properties. The IDTs pattern has been obtained by dissolving the sacrificial photoresist layer in acetone.

The following important process of the fabrication flow was the deposition of the guiding layer, whose aim is to keep the wave at the surface of the piezoelectric material, in order to increase the sensor's sensitivity. A silicon dioxide layer of 2 μm thickness has been deposited for this purpose. Due to the pyroelectric phenomenon present in the $LiTaO_3$ substrate, the SiO_2 layer has been deposited using PECVD method, which offers the advantage of low temperature deposition (200-400ºC). The acoustic wave propagation velocity in the waveguiding layer (2850 m/s) is lower than in the $LiTaO_3$ piezoelectric substrate (4112 m/s), which makes it suitable for the proposed application [9]. In addition, the SiO_2 layer deposited over the already patterned IDTs ensures the elements' isolation.

After IDTs covering with silicon dioxide, a second Cr/Au metallic film with 10/100 nm thickness has been deposited via evaporation technique, in order to define the detection area. The patterning process has been achieved using the second photolithographic mask. In the end of the fabrication process, to provide the access to the SAW device pads, the oxide that masks the corresponding areas is removed using the last photolithographic mask.

The delay line configuration is commonly used for SAW chemical/bio sensors and the device characterization is provided by vector network analyzers (VNAs). Transmission parameters (amplitude and phase) of the surface acoustic wave are considered for this purpose. Moreover, by using low temperature IC technology processes we overcome the pyroelectric effect presents in $LiTaO_3$ piezoelectric substrate and obtained a high reproducibility of the structures.

2.3 Packaging process

The packaging process followed two main directions: (i) the electrical connection of the chip to the package, and (ii) the sensor adapting to the liquid medium. The metallic package has four terminals insulated by means of glass beads supporting the terminals. This model is compatible with high frequency operation because the glass has low losses [10], and the beads are 1.5 mm long.

Connections between the chip pads and the package terminals have been realized using 25 μm diameter gold wires (a SEM detail is shown in Fig. 2.a). To use the SAW sensor for chemical sensing it was designed a sample holder consisting of a polymer body provided with a small cylindrical cavity for liquid retention and a rubber gasket for sealing the sample contact area; both the mechanical elements are chemically inert to aqueous solutions. The packaged sensor, assembled on a test fixture with microstrip transmission lines for connections to the VNA, and the liquid sample holder are presented in Fig. 2.b.

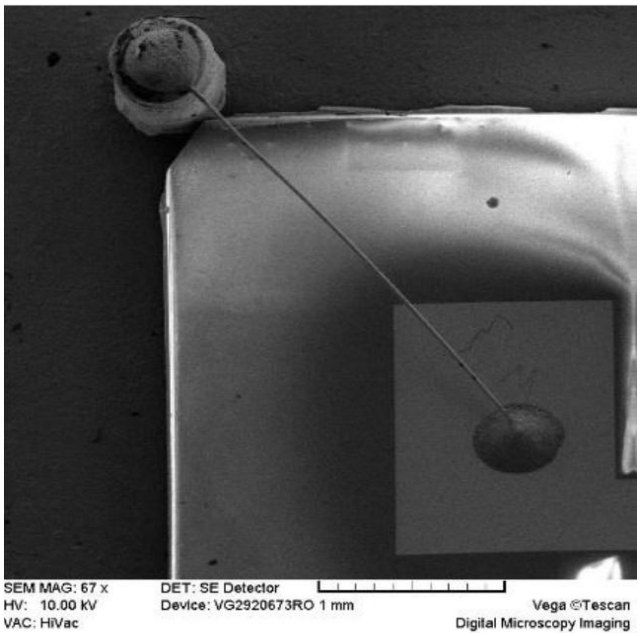

Fig. 2.a. SAW chemical SH-SAW sensor packaging: Pad connection to the package terminals.

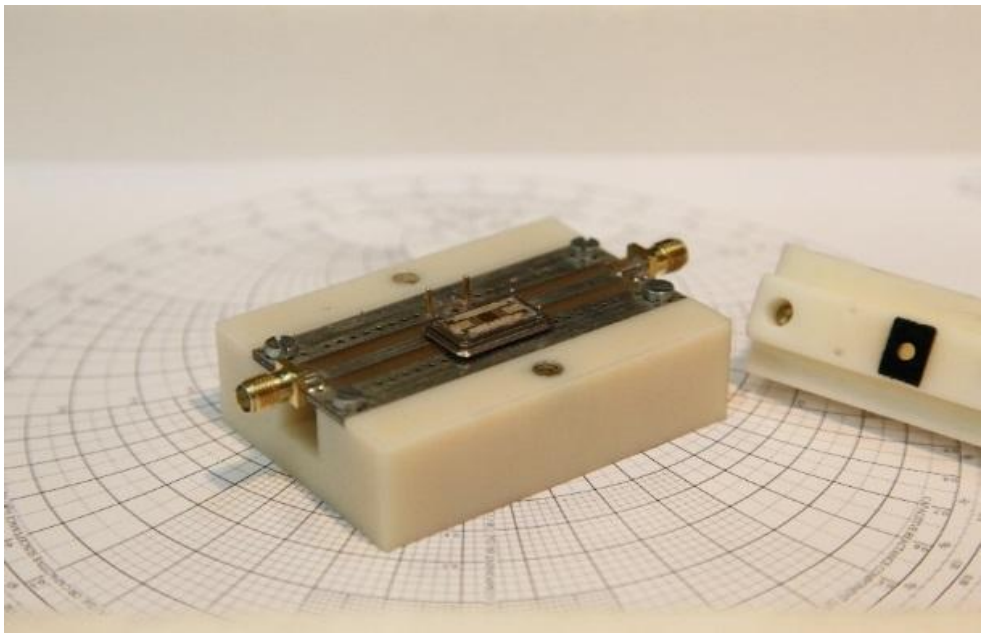

Fig. 2.b. SAW chemical SH-SAW sensor packaging:
Liquid sample positioning [11].

3. Experimental results

Accurate measurements of the transmission parameters are provided by laboratory grade VNAs, e.g. the models 37397D and MS46122A (from Anritsu) existing at IMT Bucharest. These equipment are capable to cover extremely large frequency bands with excellent resolution of the complex network parameters.
Errors due to the measuring cables, adapters, and other accessories inserted within the RF signal path are "absorbed" during calibration procedures, by means of proper calibration devices.

Before their using in measurements, the sensors must pass through a calibration procedure, required to take into consideration the effect of the material properties and manufacturing tolerances.

After RF calibration, the sensing devices are ready for regular measurements.

The calibration task is carried out under controlled conditions with respect to the sensitive layers and the state of aggregation of the substance to be detected:

- the measuring cell is filled with a reference liquid solution that does not contain substances that could interact with the sensitive layer, or
- the measuring cell is introduced into a hermetic chamber with a known concentration of the *gas* type to which the cell is sensitized.

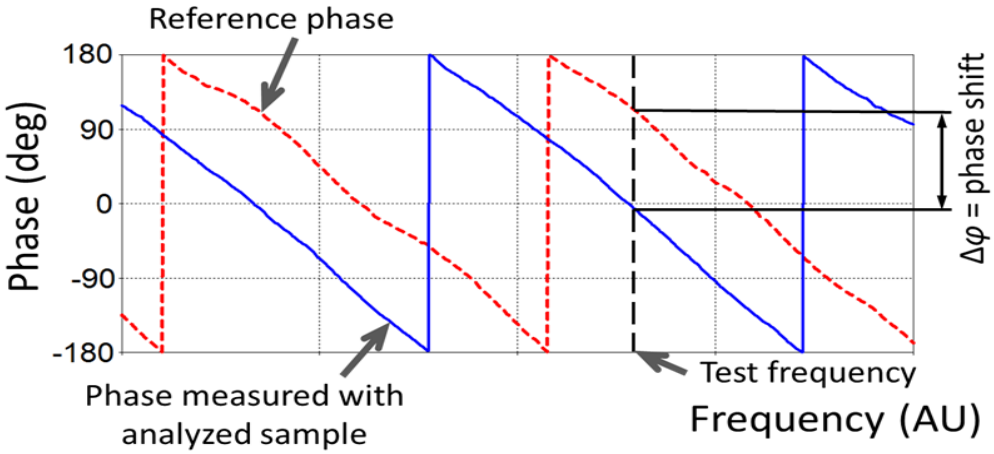

Fig. 3. Phase shift $\Delta\varphi$ of the trasmission parameter observed as phase difference between empty cell (reference value) and in presence of the analyte.

The main parameters associated to the phase of the electrical signal used during measurements are presented in Fig. 3.

The traces corresponding to the cell filled with

(i) the reference solution (*reference phase*) and

(ii) sample of controlled concentration of the analyte (*measured phase*) are compared on the same graph.

Since the phase representations have abrupt transitions for each $\pm$ 180° change-over, the test frequency has to be properly selected over the experimental graph for avoiding any misinterpretation.

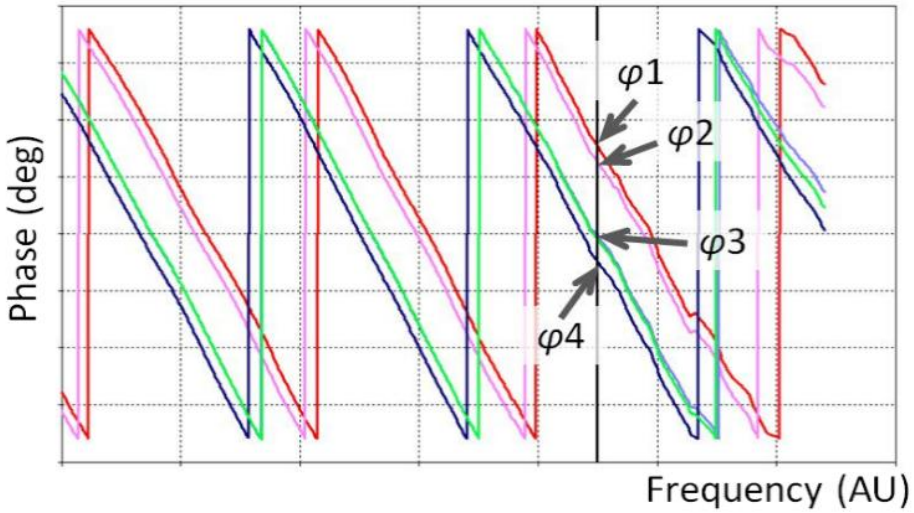

Fig. 4. Phase changes due to variable concentration of the specific analyte.

In case of cell calibration, the procedure described for a single measurement is repeated over a broad range of known concentrations, in order to cover the future experiments. The reading frequency is selected following the same rule (Fig. 4). All phase shift values $\Delta\varphi$ measured between the reference and experimental traces are used to draw the corresponding calibration graph (Fig. 5).

Once the calibration is finished, the graph is available for characterization of samples with unknown concentration according to the procedure described below:

- the phase shift corresponding to the measured sample is marked on graph's ordinate;
- a horizontal line is drawn from the actual phase shift value until it intersects the calibration curve;
- the vertical line drawn from the intersection point crosses the abscissa in the point of the unknown concentration.

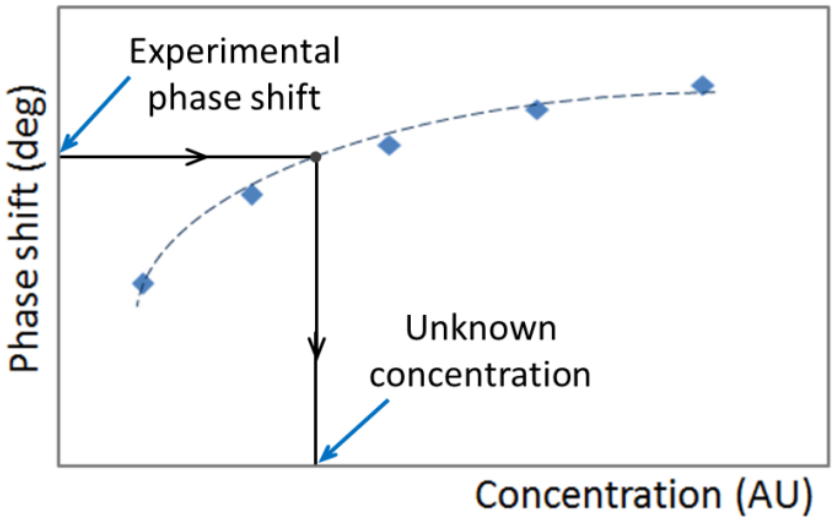

Fig. 5. Example of calibration graph based on experimental results.

This characterization method has been successfully used to determine the mycotoxin concentration in a liquid sample [8].

The VNAs are particularly useful for accurate measurements and for calibration purpose as well. However, these equipment units are bulky and very expensive for most applications. New characterization methods and low cost test fixtures were therefore developed, especially for portable units.

A low-cost measurement alternative consists in inserting the transmission-type sensor in a positive feedback loop of an amplifier (Fig. 6.a).

There are specific amplitude and phase conditions to be simultaneously fulfilled within the loop for starting the oscillation of this circuit [12].

Therefore, the operation frequency is a function of the transmission parameters within the acoustic wave path, that are dependent on actual interactions between the measured sample and the sensing area of the test cell provided with a SAW sensor [13].

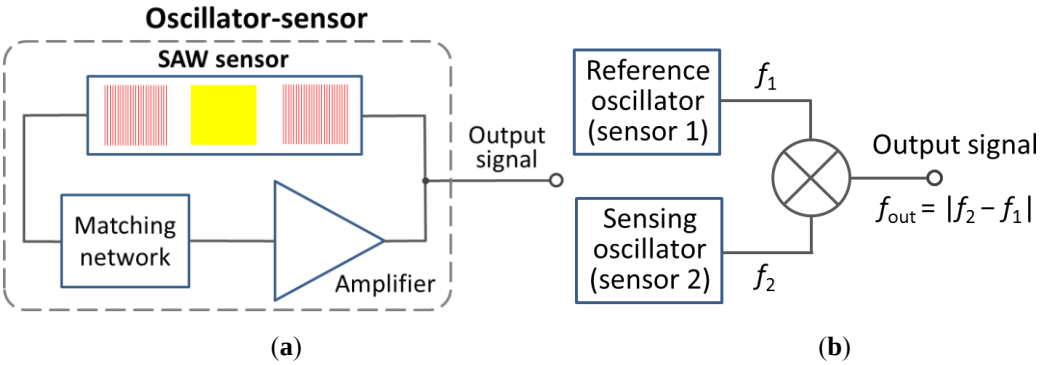

Fig. 6. Block diagram with single oscillator incorporating the SAW sensor measurement cell (**a**), and differential, dual oscillator structure (**b**).

The measurement cells are sensitive to certain environmental conditions not properly controlled during long periods of time, e.g. temperature and pressure, or directly related to the analyte.

The differential systems (Fig. 6.b) can be therefore used for compensating these "external" effects, with one cell for reference, and the other one for sensing:

- the reference sensor provides a signal which is exclusively influenced by the environmental conditions, since its sensing area is not in contact with the analyte,
- the response of the second sensor to environmental factors is similar to that of the reference sensor and, furthermore, is influenced by the concentration of the specific analyte under test.

Since the sensors are identical, they have similar behavior in the presence of the external disturbing factors; hence the frequency of the output signal becomes proportional only to the analyte's concentration.

$$f_{out} = |f_2 - f_1|$$

This setup can be used successfully to give a comparison of specific and non-specific responses, as well as to eliminate the external (environmental) effects, not directly related to the specimen under test, e.g. temperature, humidity, pressure.

Also, the differential method provides low cost measurements of gaseous and liquid analytes, because the VNA in no more required.

4. Conclusions and Future Prospects

As stated in this paper, throughout the fabrication stage of the surface acoustic wave (SAW) devices, optimal technological solutions for 36°YX $LiTaO_3$ substrate processing have been done.

These solutions are meant to value over the excellent piezoelectric properties of the substrate, but in the same time to avoid the undesired accompanying pyroelectric effect. The detecting area of the sensor was fabricated from a gold film, thus comprising a good immobilization of antibodies, while the silicon dioxide has ensured the trapping of the surface acoustic waves on the surface of the piezoelectric substrate.

A functional testing of the SAW sensor was done and it was established how the phase shift of the sensor transfer function is depending on mycotoxin solution concentration [8].

The design of a differential measurement system is considered for future, accurate measurements. Two identical delay line devices, one for sensing and the other one as reference, are required for the best performance.

This setup can be used successfully to give a comparison of specific and non-specific responses, as well as to eliminate the undesirable environmental effects (temperature or humidity variations).

In the future work, it will be attempted to improve the sensitivity of the SH-SAW chemical sensor by replacing the SiO_2 guiding layer with zinc oxide [9].

With respect to the packaging part of the devices, the cell used to adapt the sensor to the liquid medium could be replaced by microfluidic channels, designed and fabricated on the same chip with the sensor.

The structural material of the microfluidic channels could be the SU8 polymer, material that requires low processing temperatures.

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