SINGLE SH-SAW SENSOR AS A DISTRIBUTED SENSOR ARRAY FOR LIQUIDS RECOGNITION

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Abstract- Basic possibility to use single-channel shear-horizontal surface acoustic waves sensor constructed as a delay line on multilayered structure with 36° YX LiTaO₃ substrate for liquids recognition has been proposed. The changes under analytes influence of the sensor pulse responses obtained by transformation to time domain of one of the S-parameters measured in frequency domain have been used as sensor responses. Examples of responses to some liquid analytes have been demonstrated for a sensor based on LiTaO₃ – SiO₂ – molecularly imprinted polymer layered structure. Qualitative explanation of the result is given by treating such a sensor as a distributed sensor array.

Index terms: Liquid analyte, SH-SAW delay line, 36° YX LiTaO₃ substrate, wave reflections, sensor array, S-parameter, fast Fourier transform, molecularly imprinted polymer.

I. INTRODUCTION

Modern systems of chemical substances identification, whether it be «an electronic nose» or «electronic tongue», are based on so-called sensor arrays consisting of some number of separate sensors, having different but sufficient for measurement responses to different components of analyzed substances [1], [2]. Usually individual sensors in such an array are fabricated using different materials of sensitive elements or even different physical principles of operation. Surface acoustic waves (SAW) sensors also can be used in sensors arrays. In the case of electronic nose Rayleigh type of SAW are usually used in such sensors. The difference in their responses to different chemical components is provided by different selective layers covered the working surface of the SAW sensors, or can be achieved using different crystallographic orientations of substrate [3] or by some perturbations of substrate properties [4]. SAW with
shear-horizontal polarization (SH-SAW) [5] is used when SAW sensors are exploited in sensor arrays in electronic tongue system. In comparison with Rayleigh SAW the SH-SAW has no normal component of displacement and therefore it can propagate with low attenuation along the substrate surface contacting with liquid and viscous media. This feature of SH-SAW permits to construct sensors capable to work in liquids. When SH-SAW liquid sensors are used in the sensor array the difference of their responses to different chemical components is reached by using different selective layers [6] or by changing the working frequency of the sensor [7].

Plates of 36° YX LiTaO₃ as substrates and traditional for SAW devices interdigital transducers (IDT) are used for excitation and detection of SAW in majority of liquid sensors on SH-SAW at present time. Both surface skimming bulk acoustic waves (SSBW) with SH polarization and leaky surface acoustic wave (LSAW) with the same polarization are exited by IDT on 36° YX LiTaO₃ substrate. The phase velocities of these two modes on a free surface of the substrate practically are equal and effective conversion of LSAW to SSBW occurs. For weakening such a conversion the space between the IDTs on the substrate is usually coated with a conducting film, thus adjusting the waves closer to the surface. In some works (see for example [6], [7]) dielectric layer of several micrometers thick was deposited on the substrate surface for increasing the concentration of wave energy near the surface and thus increasing sensor sensitivity. Such layers serve for SH-SAW as waveguides. Thus, in reality substrates used for SAW liquid sensors can contain several areas with different electrical and acoustical properties representing discontinuities for SH-SAWs. SH-SAWs are effectively reflected from end faces and other discontinuities of a substrate. As a result, rather high ripples on amplitude-frequency and phase-frequency responses occur and when CW signals are used (as in overwhelming majority of published experimental works). For correct measurements of sensor responses (usually there are changes in amplitude and/or in phase of sensor output signal) it is necessary to undertake special measures reducing these ripples [7], or eliminating this problem of distortion at all using a pulse mode of operation [8].

In the present work the experimental results demonstrating possibility of identification some liquid analytes by means of single one-channel SH-SAW sensor based on multilayer structures on a 36° YX LiTaO₃ substrate are given. Reflected SAWs usually considered in SAW sensors as parasitic in our case are participated in forming sensor’s response. Unlike all known to us at the present time experimental works on SAW liquid sensor investigations in our work sensor’s
response is the change of the pulse characteristic of the sensor (delay line in our case) under the influence of analytes. Pulse characteristic of the sensor is obtained by backward fast Fourier transformation of one of the sensor’s S-parameters measured in frequency domain of sensor pass band.

![Figure 1. A schematic of samples under investigation](image)

1- substrate made of $36^\circ$ YX LiTaO$_3$; 2, 3 – IDT; 4 – Al layer; 5 – SiO$_2$ layer; 6 – layer of molecularly imprinted polymer; 7 – contact pads; 8 - gold wires; 9 – plastic border.

II. EXPERIMENTAL

Samples of sensors under investigation are shown schematically in figure 1. As a basis of these samples delay lines fabricated on $36^\circ$ YX LiTaO$_3$ rectangular substrate were used. SH-SAWs were excited and detected with IDTs consisting of 5 pairs of electrodes with the period of 40$\mu$m and apertures equal to 1.6 mm. The distance between the centers of the IDTs was 3.34 mm. The substrate area between the IDT was covered by 200 nm thick Al layer with vanadium underlayer fabricated by photolithography together with the IDTs. The size of structure topology together with contact pads was 2.9 x 5.5 mm$^2$, length of the substrates (in the direction of SH-SAW propagation) was 8.3 - 8.5 mm. Metalized surface of the substrate and the IDTs were covered by
3μ thick film of SiO$_2$. Over SiO$_2$ layer between the IDT a film of molecularly imprinted polymer (MIP) of 5μ of thickness was deposited. This MIP was synthesized of monomers of bisphenol-A glycerolate diacylate with imprinted morpholine as template molecules. Such MIP films we used earlier for increasing sensitivity to morpoline of gas SAW sensors [9], [10].

The schematic diagram of the experimental setup is shown in figure 2. For thermal stabilization, the samples were mounted on the working surface of a Peltier thermoelectric element (TEE) with a heat-conducting paste. The TEE working surface also contained a temperature-sensitive resistor to measure its temperature. We used commercial one-cascade TEE with a standard control circuit. It maintained the temperature of the SAW sensor within ±0.01°C in the temperature range 2–67°C at an ambient temperature of 20–40°C.

Figure 2. Schematic of the experimental setup.

1- sample with plastic border; 2 – TEE; 3 – network analyzer; 4 – PC; 5 – TEE control unit; 6 – syringe for analyte injection
High frequency input and output of the sample were connected with the correspondent ports of the network analyzer. Amplitude-frequency responses of the sample in “transmission” and “reflection” modes (S$_{21}$, S$_{11}$ and S$_{22}$ – parameters) were measured before and after injection of analytes on the sample surface. The network analyzer used in our experiments permitted to measure S-parameters in time domain also, i.e. pulse responses of the samples. Pulse responses could be obtained also by backward fast Fourier transform of measured corresponding frequency responses using PC.

Injections of liquid analytes on sample surface were performed by a syringe. Volumes of injected analytes were in the range 0.05 – 0.1 ml. Plastic borders prevented spreading of analytes outside the sample surface. An analyte was injected so that all surface of the sample with all its details, including contact pads of IDT, would be completely covered by the analyte. In this case, as our experiments have shown, responses of sensors did not depend on the volume of analytes.

Measurements of sample responses were performed in a certain time (≈10 s) after analyte injecting sufficient for temperature stabilization system to make temperature of injected analyte equal to the given temperature of the sensor surface. After the measurements the injected analyte was washed out from the sensor surface and then the sensor was heated up to 65° C and stayed at this temperature several minutes. After that, the temperature of the sensor was returned back to the former given temperature and control measurement of S-parameters without any analytes was performed. If the results of these measurements coincide with the previous results, we go to the measurement with new analyte. If not, the washing and heat treatment processes were repeated.

### III. EXPERIMENTAL RESULTS

Results on S$_{11}$- and S$_{22}$- parameters measurements at 105 MHz sensor center frequency in 60 MHz frequency band in the form of backward Fourier transform are presented in the figures 3 a), 3 b), 3 c) for one of the samples before and after injection of several analytes indicated in figure subscriptions. One can see that changes in pulse responses corresponding to different analytes are different thus permitting to distinguish one analyte from another like in electronics tongue with sensor arrays using the same algorithms. In our experiments specific parts of pulse characteristic may serve as analogs of responses of separate sensors of a sensor array. For example local
maxima corresponding to SH-SAW propagation time from reflective elements on substrate to receiving IDT may serve as such specific parts.

![Graph](image)

PS1, PS2, dB vs. t, μs

a) $P_{S_{1}}$, dB

b) $P_{S_{2}}$, dB
Figure 3. Time function of Relative power $P_{S_{ii}}$ of backward Fourier transform of parameters $S_{ii}$ measured at sample center frequency 105 MHz in frequency band 60 MHz at injection of several analytes at ambient temperature 22°C and sensor temperature 25°C.

a) $S_{11}$. Curve 1 – no analyte, 2 – ethanol, 3 – 1% - solution of morpholine in ethanol

b) $S_{22}$. 4 – vodka, 5 – cognac, – liquids with equal percent of ethyl alcohol content (40%)

c) $S_{22}$. 6 – distilled water, 7 – blood

Thus, the experimental results show that one-channel SH-SAW sensor can play a role of a sensor array usually fabricated of sensors with different sensitive elements or even using different physical principals. This may be qualitatively explained as follows. Working surface of our SH-SAW sensor has regions with different electro-physical and acoustical properties. Different properties of analytes (density, viscosity, electrical conductivity, dielectric permittivity, etc.) influence the propagation of SAW along these regions in various manners. SAWs with SH-
polarization are effectively reflected by edge planes of the substrate and by various discontinuities on its surface including IDTs. Efficiency of SAW reflections by various discontinuities also depends on different properties of analytes in various manners. As a result the total response of the sensor can be considered as if it contains a raw of partial responses of several different sensors. It is known that changes of SH-SAW sensor response is a nonlinear function of frequency and using of responses at several different frequencies of one and the same sensor is equivalent to using of several independent sensors [7]. SAWs in our experiments were excited in rather wide frequency band (not at a one given frequency), thus the sensor response in our case contains partial responses caused by continuous plurality of used frequencies.

![Graph](image_url)

**Figure 4.** On reproducibility of sensor responses. Time function of relative power $P_{S11}$ of backward Fourier transform of parameter $S_{11}$ measured at center frequency 105 MHz in frequency band 60 MHz at injections of ethanol by two times: first – black line, second in 4 hours – red line

Sensor responses in our experiments have rather complicated shape, and one could have a question about the reproducibility of these responses. Results represented in figure 4 can partly
answer this question. Two responses of the sensor to ethanol measured at equal conditions in 4 hours interval are presented in this figure. One can see that two curves (red and black) are practically coincided, so the reproducibility of the response is rather high at least in 4 hours. Responses of different samples may vary slightly (even without any analytes) due to differences in substrate dimensions, thicknesses, quality of used layers and some other reasons. In this sense the reproducibility from one sample to another is not rather high. However for each individual sample the responses to different analytes are different and are rather reproducible. When using “teaching” recognition methods elaborated for electronic tongue or electronic nose systems, which demands creation of an individual database of analytes responses for each device, such a not ideal reproducibility from a sample to sample is not of great importance.

It should be noted that no special means were undertaken in this our work to obtain high reproducibility of results and high reliability of the device.

VI. CONCLUSIONS

Thus experimental results reported in this work have shown principal possibility of application of single channel SH-SAW sensor for some analytes recognition and that such a sensor may be treated as an analogue of a certain virtual sensor array. To develop such a sensor up to practical application level additional investigations clearing out limitations of its main parameters, improving reliability and reproducibility of its characteristics are indispensible.

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