SAW Based Phononic Crystal Liquid Sensor with Integrated Periodic Microfluidic Channels

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***Abstract*— Phononic crystal sensors that have been developed in the last years proof being a promising sensor platform for different applications as liquid sensors. It has been already shown that the transmission spectrum of phononic structures depends on properties of liquids confined within a crystal. In comparison to known established sensor approaches, where sensors respond to near surface effects such as mass load, the distinguishing feature of proposed solutions is confining the liquid in resonant cavities of the phononic structure allowing determination of volumetric (bulk) properties of the liquid. Current contribution presents technological and experimental results of the surface acoustic wave (SAW) based phononic crystal liquid sensor research. The presented sensor concept integrates an array of periodical microfluidic channels into the SAW platform. Theoretical predictions underline strong demands regarding tolerances of phononic structure dimensions. As a result, the technological process of the sensor manufacturing had to be redesigned in a certain manner. Achieved results, technological challenges and solutions are described in current work.**

Keywords- phononic crystal sensor; liquid sensor; SAW sensor.

I. INTRODUCTION

Developed in recent years phononic crystal liquid sensors [1–4] vividly demonstrate advantages of this sensor approach. It is necessary to move the sensor to higher probing frequencies in order to achieve a higher sensitivity. The realization of the sensor in the frequency range of 100 MHz till several gigahertz requires application of different platforms for excitation and detection of acoustic waves. This frequency range is well developed with respect to surface acoustic waves (SAW) devices. The experimental verification of phononic crystal structures (PnC) in piezoelectric materials introduces well-known fabrication challenges. We are specifically interested in the development of SAW PnC liquid sensors. In comparison to well-established SAW sensor approaches, where the sensor effect is gained from surface effects (such as a surface mass load caused by absorption of analyte molecules in a recognition layer), the most promising feature of this high frequency sensor class is its capability to measure a sound velocity and a bulk viscosity of liquids with a precision superior to established instruments. It applies an acoustic signal amplification of a resonant liquid-filled cavity. The objectives of current research are the design of a SAW based phononic crystal sensor with at least one element being a liquid, and its fabrication with a high accuracy.

Originally, two different schemes were applied for building of the sensor, structures, i.e., holes and channels that are etched into the piezoelectric material and similar structures etched into an overlayer or a set of overlayers deposited onto the piezoelectric crystal.

Within the first approach, the etching of deep structures with steep sidewalls is required. This concept was initially developed towards SAW based phononic crystal sensor realization and was previously described in details [5]. Figure 1 shows an example of a manufactured regular phononic crystal in quartz.

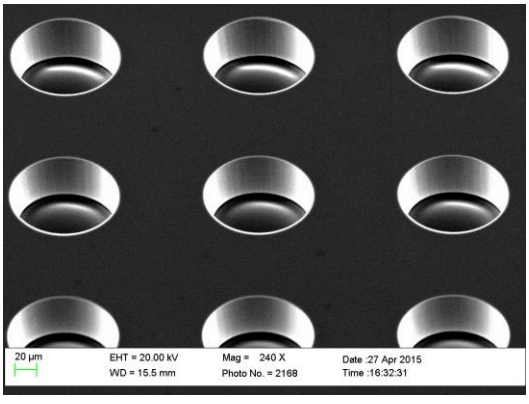

Figure 1. Etched periodical array of holes in ST-cut quartz.

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Top edges and sidewalls allow for an idealization as a hole. The known limitation lies in complexity of structuring strong piezoelectric materials such as of lithium niobate ($LiNbO_3$) or lithium tantalate ($LiTaO_3$) substrate in a quality that is required to achieve an efficient structure. [5]

However, this approach requires a piezoelectric material that contrasts some sensor requirements. We report on the development of alternative sensor approaches. As a result, the sensor concept applies polymer based microelectromechanical systems (MEMS) technology with a specific attention to an exceptional tolerances control was developed. It utilizes the structure that has been fabricated in overlayers instead of etched into the substrate. This approach allows selecting a structure material independent on the substrate. The schematic representation of the sensor utilizing this concept is demonstrated in Figure 2.

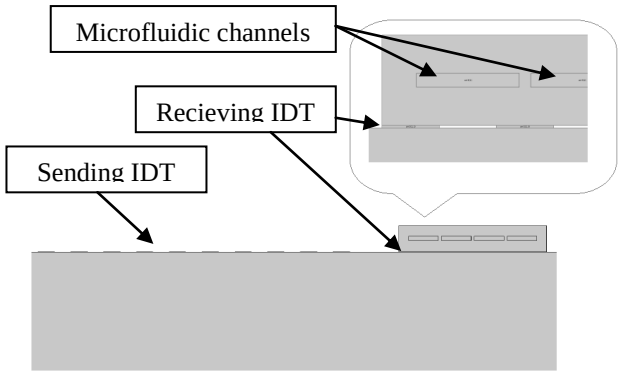

Figure 2. Schematic representation of sensor concept based on overlayer structures

The manufacture of designed structure can be completed within different approaches. The manufacturing processes can be split into a solid state based and a polymer based one. A technology based on a solid state approach introduces several advantages, such as a high long term mechanical stability, application of well-established processes etc. However, for the proposed sensor design this approach appeared to be complicated for a fabrication. On the other hand, the polymer based technological approach opens an opportunity for a rapid prototyping at a relatively low cost and allows to simplify the technological process in general. Among all possible technological processes that can be based on Polyimid, polydimethylsiloxane (PDMS), Poly(methyl methacrylate) (PMMA) or epoxy-based negative photoresist SU-8, the chosen approach must satisfy initial design demands. At the same time, the respective technology should be adjusted in a way to obtain an expected sensor structure performance.

The SU-8 negative photoresist is currently widely applied as a construction material for microfluidic devices, sensors and other applications. Due to number of advantages, such as chemical stability to most of fluids, controllable mechanical properties through the crosslinking process management, this photoresist can be applied for building of microfluidic structures for a variety of different applications [6].

Therefore, we have realized SU-8 based microfluidic structures integrated into a SAW device. A set of microfluidic channels acts as a liquid analyte container. They are periodically arranged as PnC. Several initial designs and influence of a phononic structure geometry on the sensor performance were previously discussed [7]. Despite the possibility of a further structure improvement, in current contribution we concentrate on the manufacture of designed structures.

In order to achieve a mechanical performance required for designed structures, technological parameters of SU-8 processing have to be reconsidered. Previously, an influence of technological parameters of SU-8 processing on mechanical properties of SU-8 layers have been studied [8–11]. It also was demonstrated that considering a certain application, SU-8 layer can be differently manufactured in order to fulfill defined requirements. Due to staged SU-8 processing, different improvements can be made by changing some of the conditions of certain technological steps. As it was shown in [10], the number of such technological parameters that directly influence on final SU-8 layer performance can exceed thirty. Thereby, it becomes evident that with the consideration of certain application of SU-8 structures, processing parameters have to be adjusted in order to obtain required performance.

The study of influencing of tensile properties of coated SU-8 layers has demonstrated that an increase of SU-8 curing temperature significantly changes mechanical properties of SU-8 layers [8]. It was shown that high temperature curing of SU-8 during a post exposure bake (PEB, 95 °C) and hard bake (HB, 200 °C) make layers much more fragile. Previously published results [9] have shown that changes in SU-8 processing can considerably influence following SU-8 crosslinking. It was demonstrated how a soft bake conditions, an exposure dose and a post exposure bake parameters influence on a resolution and crack formations of SU-8 layers because of inner SU-8 film stress accumulated during processing. In [10] was shown that at low soft bake temperature an exposed SU-8 polymerizes at faster rate with reduced stress. Completely crack free structures with aspect ratios of 10 and 8 for trench and ridge structures have been achieved with the soft bake temperature of 65 °C.

In order to complete closed microfluidic channels, the SU-8 adhesive bonding process should be performed. On the one hand, it is required to achieve well-defined structures. On the other hand, the fabrication process should be kept within several standard technological steps without necessity to implement complicated approaches. For this reason, the SU-8 bonding recipe has to be adjusted. In order to achieve a sufficient performance of bonding process, the polymer crosslinking reactions of bonding wafers should be taken under control. It was already shown that in order to achieve a sufficient bonding strength, it is necessary to apply the wafer with incompletely cross-linked SU-8 in order to perform a final mutual crosslinking step during the bonding process. At the same time, SU-8 should be sufficiently crosslinked to complete developing process.

In current contribution, the initial results of manufactured sensors assessment are presented. In section II, the

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technological description of sensor manufacturing process is provided in details. In section III, the information regarding the sensor experimental setup is described. Achieved experimental results and a future work are underlined in subsequent sections "Discussion" and "Conclusions".

II. TECHNOLOGY

For the fabrication of the sensor structure, following materials were utilized. Titanium and aluminum targets were used in physical vapor deposition (PVD) sputtering process and supplied by a local manufacturer. Structuring of metal layers was completed applying TI35ES positive photoresist in conjunction with TI Prime adhesive promoter that were supplied by MicroChemicals GmbH. AZ and AZ400K photoresist developers as well as acetone, isopropanol, aluminum etcher, 1-propanol and other supplies were ordered from Carl Roth GmbH and Sigma-Aldrich Chemie GmbH. Applied for structuring of microfluidic channels SU-8-50 photoresist and mr600Dev developer were supplied by Micro Resist Technology GmbH.

The deposition of metal layers was completed with LS 500 ES physical vapor deposition equipment. For photoresist spin coating, SUSS Labspin manufacturing line was utilized with SUSS_MA6_BA6 mask aligner. Processes of SU-8 adhesive bonding were conducted in SUSS SB6E substrate bonder. Manufactured SAW based phononic crystal sensors were analyzed with Zeiss EVO 50 scanning electron microscope and FTR MicroProf 300 profilometer.

The technological process of a SAW based phononic crystal liquid sensor manufacturing begins with fabrication and analysis of SAW structures. According to the sensor design, the lithium niobate ($LiNbO_3$) 128° Y-X cut wafers with X direction of the wave propagation were chosen as a substrate material. In order to excite and detect surface acoustic waves, the interdigitated transducers (IDTs) have been manufactured on the surface of the substrate.

The manufacture of phononic structure was completed atop of fabricated IDTs. The phononic structure represents a system of periodically placed microfluidic channels closed from the top and the bottom with another polymer structure. In current work, the microfluidic structure and covering (interfacial) layers were fabricated utilizing SU-8 polymer. The structure manufacturing process involves subsequent layer after layer processing. In order to complete the structure, the wafer should be processed with two structured SU-8 layers. Initially interfacial SU-8 layer has to be defined atop of the IDTs area and then the microfluidic channels should be structured atop.

An application of polymer based bonding technology was utilized for manufacturing of a covering layer. The covering layer was fabricated on a silicon wafer basis and then transferred to the structure. The omnicoat releasing layer was utilized to remove the handling wafer. SU-8 50 covering layer was then spin coated and soft baked at the temperature 65°C during 3 minutes. Thereafter, the temperature was ramped up to 85°C at which the wafer was baked another 10 minutes. At the completion of the soft bake, the wafer was cooled down back to the room temperature during 40 minutes. Then, fabricated SU-8 layer was exposed with an exposure dose of 160 mJ/cm^2 in SUSS_MA6_BA6 mask aligner. The post exposure bake of the covering layer was made at the temperature of 65°C during 2 minutes with following ramping up to 85°C where it was baked another 10 minutes and then cooled down to the room temperature during 40 minutes. After a completion of the covering layer, another thin layer of SU-8 5 that serve as an adhesive during bonding process was spin coated. As soon as the layer is pilled off, it was removed and rinsed in deionized water (DI-water). Afterwards, dried covering layer was applied for bonding process.

The bonding process starts with evacuating of the camera. During the preheating step at the temperature of 45°C during 3 minutes, the rest of the surface contaminations and SU-8 solvents supposed to be removed. After that, the structure was top loaded with a pressure of 1000 mBar and heated up to 50°C. At this temperature, the structure was bonded during 10 minutes and then slowly cooled down back to the room temperature. Thereafter, bonded with covering layer wafer was exposed with an exposure dose of 160 mJ/cm^2. Afterwards, it was post exposure baked at the temperature of 65°C during 2 minutes and 85°C for 10 minutes with following cooling down back to the room temperature during 40 minutes.

Completed example of SU-8 microfluidic channel is demonstrated in Figure 3.

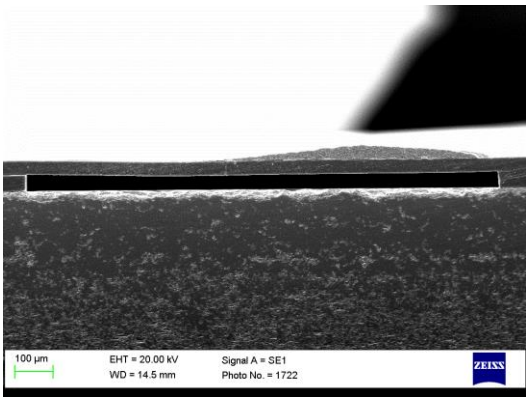

Figure 3. Cross section of the manufactured microfluidic channel.

III. EXPERIMENTAL

Experimental investigations of the manufactured sensor were completed with the help of probe station SUSS EP6 with high frequency probes, Figure 4. A complete 4" LiNbO3 128° Y-X cut wafer with IDTs and integrated microfluidic structures was placed on a probing table. High frequency probes (2 signal probes and 2 ground probes) were connected with a help of 50 Ohm up to 500 MHz coaxial cables with network analyzer in S-parameters measuring mode. The structure was connected as a standard 2-port SAW device. Analyzed liquid was manually placed in microfluidic structure with a micropipette. Transmission (S_{21} parameter) amplitude and phase response were measured with Agilent4395A together with an S-parameter test set Agilent 87511A (100 kHz–500 MHz). Several subsequent

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measurements were performed to reduce a measurement error. The repeatable sensor response was recorded for each measuring liquid mixture.

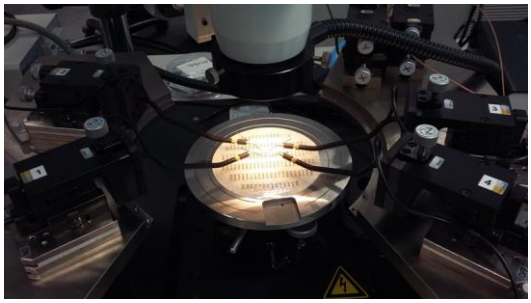

Figure 4. Experimental setup.

Measurements were conducted directly on the wafer (4" LiNbO3 128° Y-X cut wafer) with fabricated microfluidic phononic structures and IDTs.

Measurements were completed with binary mixtures of a deionized water and a 1-propanol in molar concentrations (X) range 0.23 – 0.507 that corresponds to the range of the most linear speed of sound dependence. The respective speed of sound and density data for water – 1-propanol mixtures are shown in Table 1.

TABLE I. PROPERTIES OF WATER AND 1-PROPANOL MIXTURES [12].

1-Propanol molar concentration, X	Density, kg/m^3	Speed of sound, m/s
Water	998	1483
0.158	933	1472
0.230	908	1421
0.347	881	1367
0.507	852	1322
1-propanol	804	1220

IV. RESULTS

As it can be seen, the amplitude and phase measurement results provide a distinct sensor response on different liquids that filled into the microfluidic channels Figure 5-6.

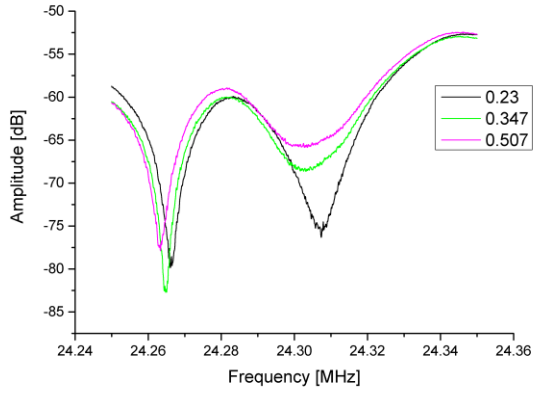

Figure 5. S_{21} parameter (transmission) amplitude response for binary mixtures of water and 1-propanol with molar concentration of 1-propanol X = 0.23; X = 0.347; X = 0.507

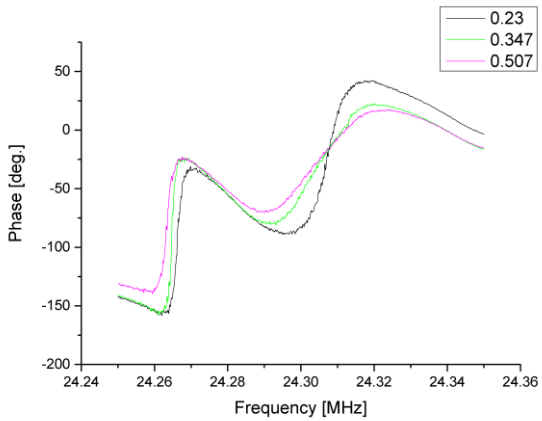

Figure 6. S21 parameter (transmission) phase response response for binary mixtures of water and 1-propanol with molar concentration of 1-propanol X = 0.23; X = 0.347; X = 0.507

The amplitude minima as well as the corresponding phase shift demonstrate a tendency to move in a direction of lower frequencies when the microfluidic channel is filled with a liquid with lower speed of sound. The dependence of phononic crystal sensor response on speed of sound of the liquid constituting the PnC or filling the defect (such as cavity) were previously demonstrated [2, 4, 13, 14]. It has been shown that frequency of corresponding transmission peak (or dip) depends mostly on changes in speed of sound of the liquid analyte rather than on changes in density or viscosity. However, in the reported case, it should be noted that transmission curves have two clear transmission minima that behave differently, Figure 5. The (local) transmission minimum frequencies depend on the molar ratio of 1-propanol in water in a similar manner; however, the band width of the second one is much broader than the first one. Both, band width and amplitude change considerably with molar ratio. As characteristic for a dissipation mechanism, bandwidth decreases with an increasing depth of the dip in the spectrum. We assume that the second minimum corresponds to a complex vibration of the polymer structure with different contributions from the microfluidic liquid resonator and overlayers and radiation or mode conversion losses.

The phase shift results match to the transmission amplitude response, Figure 6. It can be seen that the first phase shift corresponding to the first minimum is much sharper than the second one. Although we note a slight difference in slope we find a shift to a lower frequency when microfluidic channels are filled with liquids with decreasing speed of sound for the first transmission minima consistent with the amplitude findings. The phase corresponding to the broad band minima crosses at a frequency of about 24.31 MHz. However, this finding is also just a consequence of a change in wave attenuation.

The physical understanding is subject of ongoing research, specifically the analysis of the involvement of complex vibration mechanisms and their interaction with waves.

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V. CONCLUSIONS

The alternative approach for phononic crystal based sensors has been demonstrated. The SAW sensor platform serves as a reliable sensor platform for frequencies of several tens megahertz with good perspectives in a range to several hundreds of megahertz and perhaps up to a few gigahertz, very much depending on accuracy issues in fabrication of the downscaled feature sizes. In comparison to traditional SAW sensors, the phononic crystal SAW sensor can be applied to liquids and is sensitive to volumetric material properties such as speed of sound of an analyte. Our alternative overlay technology approach can be considered as a replacement of the technologically challenging approach that is based on realization of PnC structures directly in piezoelectric substrates. The experimental results demonstrate the feasibility of described approach. It provides a distinct and predictable sensor response on speed of sound of binary mixture of water and 1-propanol.

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