Sensors and Actuators B 196 (2014) 272–281

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb

A novel approach for differentiation of liquid samples with surface acoustic wave transducers and embedded microcavities

Sukru U. Senveli [a,c,*], Onur Tigli [a,b,c]

[a] Electrical and Computer Engineering, University of Miami, Coral Gables, FL 33146, USA
[b] Department of Pathology, Miller School of Medicine, University of Miami, Miami, FL 33136, USA
[c] Dr. John T. Macdonald Foundation, Biomedical Nanotechnology Institute at University of Miami, Miami, FL 33136, USA

ARTICLE INFO

Article history:
Received 29 October 2013
Received in revised form 31 January 2014
Accepted 3 February 2014
Available online 12 February 2014

Keywords:
Surface acoustic waves (SAW)
Rayleigh waves
Acoustic and fluid interaction

ABSTRACT

We discuss a novel method for sensing and differentiation of analytes trapped in a microcavity with an emphasis on liquids. The proposed sensing mechanism relies on capturing the analyte of interest in a microcavity etched on the delay line in contrast to the conventional mass loading method. The structure mainly consists of input and output interdigitated transducer (IDT) electrodes in an otherwise standard delay line configuration operated in Rayleigh mode along with a microcavity etched between the IDTs to trap minute amounts of liquids. Firstly, the responses of the system with the microcavity are explored using finite element method (FEM) analysis. Then, experimental results from delay lines on two different substrates, namely, Y-Z lithium niobate and ST-X quartz are analyzed. The system can distinguish between liquids with glycerin concentrations ranging from 60% to 90% in water and less than 5 pL in volume in the high frequency range of 197 MHz and 213 MHz based on frequency and phase shift readings. Lithium niobate samples with 1.2 μm deep microcavities provide an overall frequency sensitivity of −7.7 kHz/(% glycerin). Quartz samples with 8.5 μm deep microcavities have a sensitivity of -0.13°/(% glycerin). The minimum density–viscosity product experimentally differentiated using embedded microcavities is $1.9\,\text{kg/m}^2\sqrt{\text{s}}$. It is concluded that this method can be used to trap and interrogate minute amounts of liquids with different properties. Experimental results demonstrate that our approach can possibly be extended to certain solids, and to more complex structures like single biological cells.

1. Introduction

Surface acoustic waves (SAW) are a form of acoustic waves that travel along the surface of a medium. Their properties are altered to great extents upon interaction with irregularities along their path of propagation; therefore, they make excellent candidates for sensors. Traditionally, acoustic wave sensors have been studied extensively and used in fields as diverse as temperature, torque, and viscoelasticity measurements in various modes such as pressure, mass loading, and viscosity [1,2].

SAW gas sensors and biosensors are generally used in mass loading mode where the added acting mass of the delay line medium or any particulates captured in the delay line portion of the system causes a drop in the resonance frequency of the filter configuration [3–5]. Surface functionalization coatings and absorbent films usually aid the capturing process in the case of biosensors. Rayleigh waves are one of the most commonly used types of surface waves. The fact that their energy is contained close to the surface makes the device surface very sensitive at the expense of the capability of immersed operation due to extensive loading losses from liquids. On the other hand, Lamb wave, Love and other SH-wave based devices have been used for liquid based measurements and specific measurements such as viscosity [6–8]. As a result, research trends moved in the direction where Rayleigh mode SAW sensors did not find much use for integration with liquids. Still, some attempts have been made in the past where large masses of liquids were used with bounce and waveguide modes for coupling with Rayleigh waves [9]. There have been plenty of studies toward acoustic streaming of liquids with or without particles and acoustic processors [10]. Two things are common in most of these studies. Firstly, amounts of liquid being used are quite large, causing complete conversion of SAWs to acoustic compressional waves in liquid thereby eliminating the response due to SAWs on the receiving end. Secondly, such wave interactions have generally been considered as manipulation or actuation tools as opposed to sensing tools with some exceptions [11].

* Corresponding author at: Electrical and Computer Engineering, University of Miami, Coral Gables, FL 33146, USA. Tel.: +1 305 284 6055.
E-mail addresses: senveli@umiami.edu, tigli@miami.edu (S.U. Senveli).

http://dx.doi.org/10.1016/j.snb.2014.02.007
0925-4005/

The approach we outline in this study differs from the previous studies outlined above in both categories, and uses *SAW–liquid* interactions as a sensing and differentiation mechanism for minute amounts of material. In the past, the only similar research on a similar topic with embedded microcavities has been a theoretical study about insertion of rectangular prism shaped polystyrene plugs into the delay line of SH-wave sensors with the purpose of not sensing but improving transmission characteristics and loading sensitivity [12]. No other research exists on SAW interactions for interrogation of minute amounts of matter in a similar way to our approach to the best of our knowledge. Here we present a novel sensing mechanism in which a material, in this case, a very small amount of liquid, placed inside a microcavity etched on the delay line can be interrogated using Rayleigh type surface waves. The mechanism differs from standard delay line configurations as an embedded microcavity which breaks the delay line is introduced. This structure causes dispersion and loss of a portion of SAW to bulk waves depending on the geometry but the resultant output transfer characteristics are dependent on the way the surface wave interacts with the material trapped in the microcavity. It is aimed to show that the properties of minute amounts of samples can be interrogated using this approach. During the course of this paper, first we give the overview of the model we use for the system followed by finite element method (FEM) analysis. After detailing the microfabrication procedures, experimental techniques are discussed. Finally, measurement results are presented.

2. Modeling and simulations

The study of piezoelectric–liquid interface has been carried out in the literature before, particularly in the discussion of acoustic streaming applications. In the commonly studied versions of this setup, the interface between the piezoelectric layer and liquid droplet is only on the lateral plane where a liquid droplet remains on the surface of the piezoelectric layer with a contact angle given by the hydrophobicity/hydrophilicity of the substrate. After hitting the boundary, the Rayleigh wave decays rapidly and the transverse component emits longitudinal waves into the liquid at a certain angle. This critical angle is defined in an analogous fashion to Snell's law, where it is defined in terms of the ratio of the wave velocities in the two media [13]. On the other hand, detailed solutions and exact analytical expressions for Rayleigh waves can be found in [14]. In our case, a similar approach is used, however, there is a microcavity filled with a liquid in the delay line and the surface wave penetrates into it according to a pressure based acoustics model. This acoustics model treats the pressure propagating inside the liquid according to a wave equation. An overview of the domains involved is shown in Fig. 1(a). A simplified 2D version of the actual problem to be solved is depicted in Fig. 1(b). Simulations were carried out using commercial finite element method (FEM) simulation package, Comsol Multiphysics. The simulation efforts can be broken down into three sections. Firstly a regular delay line is modeled to form a base model for the SAW IDTs after the correct surface wave mode has been identified. The defining characteristic of SAW behavior, insertion loss information, is obtained. Following this, response with an empty microcavity placed in the delay line is obtained. Then, simulation results where the microcavity is filled with different liquids are presented.

In the simulations, the liquid has been modeled as a Newtonian fluid defined by its density, characteristic speed of sound, and viscosity. The simulation domains cover the contact of liquid and the piezoelectric surfaces which are connected to each other via appropriate boundary conditions. Also, it is assumed that there is no slip associated with the liquid interface. The interface between *piezoelectric–air* domains and *liquid–air* domains can be considered as a soft boundary for acoustic waves where the pressure is zero as the acoustic transmission from relatively high acoustic impedance media to very low impedance medium such as air can be safely neglected.

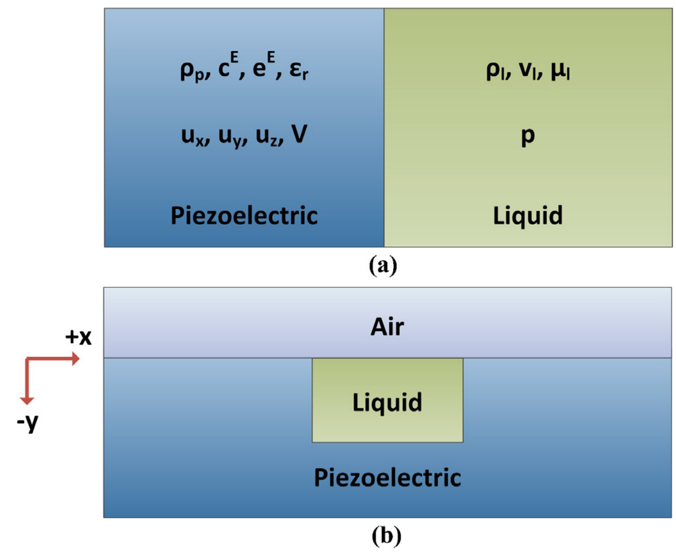

Fig. 1. (a) A schematic representation of the important domains in the model. The characteristics of the media and independent variables are indicated. (b) Depiction of the 2D version of the real problem to be solved with the cavity. The air–liquid and air–piezoelectric boundaries can be simplified and neglected due to negligible acoustic transmission coefficient between these two pairs of media, hence eliminating the air domain completely.

The Rayleigh wave mode is found around 215 MHz for Y-Z lithium niobate and 204 MHz for ST-X quartz using harmonic analysis to serve as a basis for further frequency domain simulations. Wave velocities are calculated as 3446 m/s and 3259 m/s, respectively which are close to the expected and tabulated phase velocity of these waves on the Y-Z lithium niobate, 3488 m/s, and on ST-X quartz, 3158 m/s. The 2-D simulation of the system can provide insight into the behavior and coupling of the piezoelectric and liquid domain, as long as the microcavity is large enough or the aperture is small enough (in the out-of-plane direction) so that they overlap. If the microcavity and aperture are close in size, the effects of non-uniformity along the out-of-plane axis which gives rise to diffraction artifacts can be ignored. In simulating the system, we use constant overlap electrode pairs and unity metallization ratio to simplify the designs. The output insertion loss characteristics of the system can be obtained in one of two ways: directly from frequency domain simulations or by applying Fourier transform to the time domain results to convert them to frequency domain data (which, in practice, is applying discrete Fourier transform to a good approximation). The frequency domain solutions were preferred over time domain solutions in general as they are less time consuming and they allow for direct examination and evaluation of the exact wave behavior at a given frequency as shown in Fig. 2.

The IDTs in the simulations contain 64 electrode pairs, whereas the electrode width and spacing are both 4 μm yielding a wavelength of 16 μm. This wavelength is selected since it is comparable to the microcavity diameter. The number of electrode pairs yields a sharp enough passband peak to comfortably observe the output while keeping the simulated system size manageable. On the other hand, the microcavity diameter is selected to be 24 μm or 1.5λ as it is easy to work with and also this size allows for further testing of similar sized objects in the micro scale for future studies. It is also an easier task to fabricate actual SAW microcavity sensors with corresponding feature sizes. A design trade off in the proposed devices is the size of the aperture. As the aperture is made smaller,