Hexagonal Surface Acoustic Wave Devices for Enhanced Sensing and Materials Characterization

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Abstract

We present the design, fabrication and testing of a hexagonal surface acoustic wave (SAW) array device fabricated in YZ lithium niobate for non-destructive evaluation of thin organic, inorganic and biological films. Propagation along the Y-axis generates a Rayleigh mode wave where off-axis propagation excites a mixture of other SAWs. Our approach permits rapid and simultaneous extraction of multiple film parameters (film material density or thickness, Lamé and shear moduli, sheet conductivity) of a thin film material to achieve a more complete characterization than when a single SAW device is utilized. In sensor applications, this capability translates to better discrimination of the analyte and possibly more accurate quantification. The device is based on a double split finger delay-line design with a line width of 4 μm and a delay path of 197 λ. The individual delay paths of each hexagonal device intersect in the center of the die producing a single region for sensor analysis. Additionally, the central region where the acoustic waves intersect is shorted to reduce the number of modes of waves traversing the surface. Initial testing has shown the pass band frequency of the individual delay paths to be centered around 97 MHz. The acoustic velocities of the rotated device have been measured to be 3593 m/s, 3721 m/s, and 3620 m/s, which correspond to the theoretical values of 3542 m/s, 3646 m/s, and 3622 m/s, respectively. Vapor sensing tests were conducted by exposing a poly(isobutylene)-coated device to various concentrations of benzene, chloroform, and n-hexane in the range of 0.8 to 16.6 volume percent. Measured attenuation and phase angle shifts at a fixed, near-center frequency revealed significant, signature-type differences for the three delay-paths at each exposure concentration. These responses can be exploited in constructing better sensors and sensor arrays utilizing these hexagonal SAW devices.

Introduction

For many years, SAW devices have been used both individually and in arrays as sensors and for materials characterization in a variety of applications ranging from gases/vapors to biological systems [1-3]. Within this broad range of uses for SAW devices, is the need for non-destructive testing of thin films. Current technology is based on using dual delay-line configuration with one delay-line used as a reference to compensate for environmental changes [2]. For basic sensor applications, this technique is sufficient; however, it is possible to design simple devices that can achieve better sensor characteristics as well as materials characterization possibilities utilizing simple device response models derived from perturbation theories [2]. The hexagonal SAW device presented in this work is one such example that affords the possibility of extraction of multiple film parameters from responses of the three delay-lines that probe a common region. It is hoped that this and similar devices can serve as in-situ characterization tools in thin film physical and chemical deposition equipment, and perform better than the ubiquitous quartz crystal microbalance, which yields film thickness information only. It is conceivable that under specifically optimized conditions, devices or arrays of devices presented here can be relied on to monitor deposition processes in such equipment.

In sensor applications, multiple parameters extracted from the film can be thought of as multiple calibration curves which allow for a more unique characterization of the type and concentration of the analyte being sensed. Combined with the array concept, significantly more information can be obtained to better analyze the analyte. Also, many acoustic wave devices are specific to the phase in which they operate; for example, the successful Rayleigh wave device for vapor sensing is useless in the liquid phase due to excessive attenuation [4, 5]. The multiple directions in which the waves are launched in the hexagonal device of this device work are different in character, and will allow for a common device to be functional in both gas and liquid phases. In recent work, we have shown that Rayleigh wave devices can be utilized in acoustic cleaning of non-specifically bound proteins in biosensor applications [6]. With the possibility of launching shear horizontal SAW waves in one direction and Rayleigh waves in another, the hexagonal device may well serve as a better biosensor element for liquid phase applications. Such investigations are underway in our laboratory, with designs implemented in more suitable piezoelectric materials, than the lithium niobate utilized in this work.

The results of vapor sorption by poly(isobutylene) (PIB) of this work can be interpreted by known sensor response models for SAW device perturbation by viscoelastic films [7]. The response of a typical SAW sensor to an external perturbation from a viscoelastic film can be expressed by equation 1, which is independent of type of surface wave. For many determinations, it is convenient to assume that the material properties contained in the β_i and M_i terms remain constant [3, 7].

$$\frac{\Delta\gamma}{k_o} = \frac{\Delta\alpha}{k_o} - j\frac{\Delta v}{v_o} = \sum_{i=1}^{3}\frac{c_i\beta_i M_i}{\omega}\tanh(j\beta_i h) \tag{1}$$

$$R = \frac{Afv_o\rho h}{|G|} \tag{2}$$

For a thorough derivation and discussion of the use of these equations, see the work of Martin *et. al* [2, 7]. Simplifications to this equation can be made by assuming the film to be acoustically thin, and not displaying viscoelastic properties. Roughly, when R<<1 in equation 2 the polymer film is considered to behave as an acoustically thin film; whereas, when R≥1 the film behaves as an acoustically thick film [2, 7].

Hexagonal Device Design

Several IDT designs have been tested on the route to devices having linear phase, low noise, and low insertion loss. All of the devices designed and tested have been laid out using a standardized bonding pad design to increase the ease of probing while on wafer. The overall die size is a 20 millimeter square. Rotated about the center of the die are three identical bi-directional SAW delay paths consisting of an aperture of 47 λ, a delay path of 197 λ, a minimum feature size of 4 µm, with the delay path shorted. The first patterns tested consisted of a standard double split finger design with 60 finger pairs, designed to have a narrow passband. Subsequent and current designs have considerably fewer fingers. The first technique to improve the SAW filter sensor characteristics

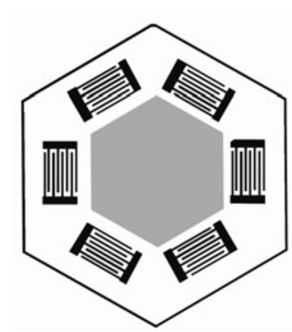

Figure 1. Hexagonal SAW device schematic

was to minimize the number of IDTs. In the second techniques, the 60 pairs were reduced by pruning to create a ladder structure. The final design used employs a weighting technique of using one regular finger followed by a split finger to make up the pair [8, 9]. A standard metallization procedure of 100 nm chromium followed by 700 nm gold was used for all of the devices. The overall schematic layout of the device is shown in Figure 1.

Electronic Characterization Methods

The hexagonal SAW devices were tested using an Agilent 8753ES S-parameter Network Analyzer connected to Mini-Circuits ZASWA-2-50DR switches. The switches were configured to allow measurement of all three delay paths without the movement of the probing fixture. The fixture consists of a custom fabricated acetal housing holding spring pins (Ostby Barton Pylon) in a mating pattern to the bonding pads of the devices. Due to the nature of the SAW devices, the hexagonal pattern resulted in the ground and signal pins to alternating, providing better than average signal properties when compared to other in house designed SAW's and fixtures. Measurement of the wave velocity was achieved using the VNA's built in transform function. This function is a Fourier transform that takes the standard S_{21} parameter and converts it to the time domain which shows the fundamental and higher harmonics of the SAW device. While in this measurement domain, the maximum value on the plot is selected; this corresponds to the fundamental frequency of the SAW device. Further analysis such as quickly applying gating to see the effects of bulk acoustic waves, triple transient effects, and internal IDT reflections of the different SAW devices was done while using the transform function.

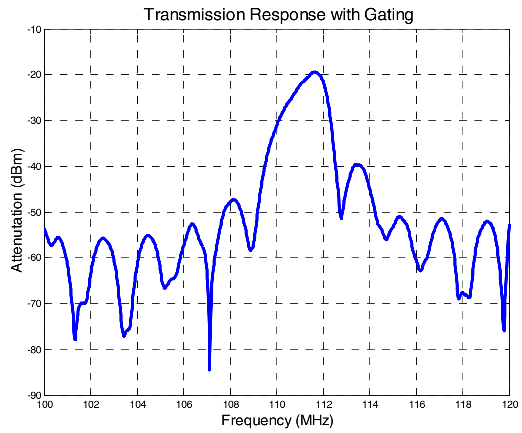

Figure 2. S_{21} measurement of one of the delay paths with gating applied having gating start and stop values of 1.218 μs and 3.878 μs

Polymer Solvent Experiments

The polymer poly(isobutylene) (PIB) was used for preliminary testing of the functionality of the hexagonal SAW devices. Tape was used to mask off the IDTs to prevent excess attenuation. A 0.5 weight percent PIB chloroform solution was used with a Badger® airbrush to apply thin films from 200-600 nm onto the SAW delay path. Following the coating, the polymer was annealed for 20 minutes.

The hexagonal SAW was connected to a homebuilt organic vapor dilution system capable of delivering four different organic vapors with

Table 1. Concentration (volume percent) of solvents in nitrogen carrier stream in volume percent

Stage	Benzene	Chloroform	Hexane
1	0.8	1.9	1.4
2	1.6	3.8	2.8
3	2.4	5.6	4.2
4	3.2	7.4	5.5
5	4.0	9.0	6.8
6	4.8	10.7	8.0
7	5.5	12.2	9.2
8	6.3	13.7	10.4
9	7.0	15.2	11.5
10	7.7	16.6	12.7

computer controlled accuracy. This dilution setup is described in detail by Upadhyayula *et al*. [10]. In this work the solvents benzene, chloroform, and hexane were used. The programmed exposure pattern consisted of a 1200 second purge followed by 600 second exposures and 600 second purges in increasing concentrations of solvent. The volume percentages of the solvent vapor in nitrogen are given in Table 1.

Results: Characterization of Devices

For SAW device characterization, the primary analysis is the transmission s-parameter (S21). From this measurement, the amount of power transmitted through the delay line band pass filter SAW is plotted against a frequency sweep. Further benefit from analysis was found by applying gating to the response to see the effects from triple transit and bulk effects. Gating was applied to remove faster and slower effects from around the fundamental harmonic while monitoring the time domain.

Table 2. Concentration (volume percent) of solvents in nitrogen carrier stream in volume percent

Orientation Euler angle (φ, θ, ψ)	Theoretical (m/s)	Measured (m/s)
(0,90,91)	3542.06	3593.30
(0,90,151)	3646.81	3721.85
(0,90,31)	3622.59	3620.73

The resulting typical S_{21} response after applying gating to remove these unwanted effects is shown in Figure 2. Calculations for velocity were made while performing the analysis for gating while in the time domain. As shown in Table 2, the measured values for the different delay paths correspond well to the theoretical velocities calculated by the Campbell and Jones method for the lithium niobate substrate.

Results: Polymer Sorption

The hexagonal SAW device response to varying concentrations of n-hexane from exposures to the polymer film is shown in Figure 3. The 500 nm PIB film on the hexagonal SAW responded to n-hexane at varying concentrations with differing attenuation and phase changes for the three delay paths. The varying attenuation of the device is an indication that the polymer film was behaving as an acoustically thick film. Upon calculation of *R* it is found that the film does fit the guideline for a thin film.

Similar results were found for the solvents benzene and chloroform. Notice in Figure 3 the response of the on-axis phase initially increases, but as the concentration increases, the phase response begins to shift in the opposite direction. The cause of the change is that the polymer film is

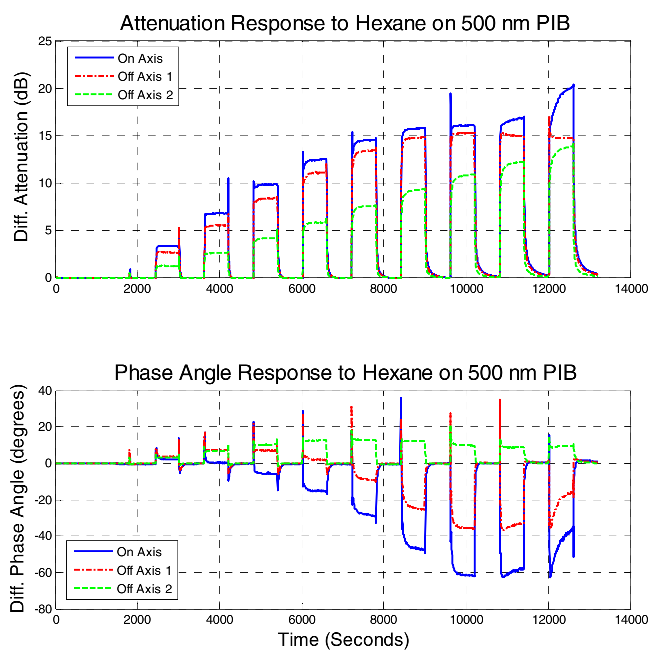

Figure 3. Difference measurements from 500 nm PIB on the hexagonal SAW absorbing n-hexane in nitrogen at 25 ºC, of volume percentages 1.5-13.1

acoustically thick and at the higher solvent concentrations, the film moduli actually can change significantly [2]. As a second means of analyzing the data, the attenuation is plotted as a function of the phase. See Figure 4.

These vapor sorption data indicate that different wave types are likely propagating in the different delay path directions of the hexagonal device. In this case of YZ lithium niobate substrate, the on-axis wave is predominately a Rayleigh wave, but on rotation about the center origin, different modes of propagation are found. Because each delay path has its own unique frequency and wave type, multiple sets of equations can be solved to extract significantly more information on the coated film than if one device were to be used, providing a fuller picture of the film properties.

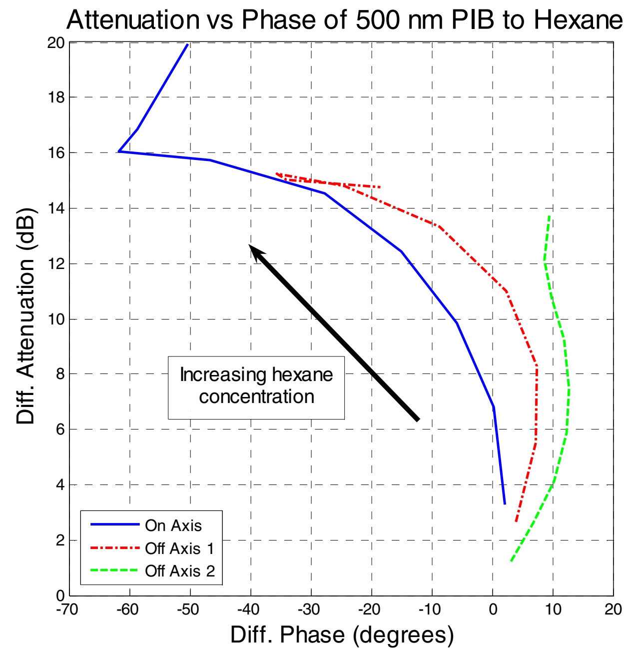

Figure 4. Relationship plot of attenuation and phase angle of 500 nm PIB on the hexagonal SAW absorbing n-hexane in nitrogen at 25 ºC, of volume percentages 1.5-13.1

Conclusions

The use of multiple SAW modes can provide more information about a sensing film as compared to using multiple SAW sensors with a single wave mode. Additional benefits of interrogating one uniform film can be realized, by providing multiple measurements for verification of responses. The use of a hexagonal pattern SAW on $LiNbO_3$ makes efficient use of the substrate, while having the potential to yield a clearer picture of the interactions of the sensing films with an analyte.

Acknowledgments

This work was funded by NSF-IGERT Grant No., DGE-0221681, by NASA-Glenn, and the MESA Institute of Sandia National Laboratories. We would like to thank Ms. Nancy Saldanha and Dr. Don Malocha of the University of Central Florida for assistance with fabrication, Mr. Randy Williams for assistance with the organic vapor dilution system, and Mr. Bernard Batson for administrative assistance at University of South Florida.

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