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Fabrication and Performance of a Surface Acoustic Wave Resonator Based Chemical Sensor

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Abstract

The fabrication and performance of a SAW resonator based chemical sensor designed at center frequency of 500 MHz is described for detection of Sarin (DMMP). The resonator and reflector grating element width (critical dimension) was 1.69 micron. The mask was fabricated and subsequently, the SAW resonator was fabricated on ST-Quartz substrate. The performance of resonator demonstrated resonance at 505.7 MHz (insertion loss within 6 dB). The sensing curve of the prepared SAW sensor with exposure to 3 ppm DMMP vapours showed a frequency shift of 700 Hz (233 Hz/ppm).

Keywords: *Chemical Sensor; DMM; Resonator; Sarin; Surface Acoustic Waves.*

1. INTRODUCTION

Acoustic wave devices have been in commercial use for a long time. Telecommunication industry is the largest consumer. Several applications of Surface acoustic waves (SAW) devices are for sensor application as torque and tire pressure, chemical sensors, vapor, humidity, temperature and mass sensor (Drafts, 2000). Advantages of SAW sensors are their low cost, ruggedness, sensitivity, reliability (Murugan *et al.* 2014). Some have the capability of being passive and wireless interrogation.

SAW devices can be used to monitor/sense gases and organic solvents, if these are coated with a material which selectively adsorbs molecules from air (Wohltjen 1984; Nieuwenhuizen 1991; Fischeraner 1995; Avramov *et al.* 2000). Use of SAW devices for detection/sensing of Chemicals/explosives/chemical warfare agents (Sarin through DMMP) has been an active area of study (Wang *et al.* 2006; Wen *et al.* 2007; Joo *et al.* 2007; Yadong *et al.* 2009; Liu 2011; Islam 2012; Afzal *et al.* 2013; Liu *et al.* 2014; Kim *et al.* 2014). In a SAW based chemical sensor, either a delay line or a resonator is inserted into feedback loop of a two port oscillator, whose frequency then depends on the number of molecules adsorbed. A delay line is the simplest structure made of two inter-digital transducers (IDT). For IDT, located sufficiently far apart, the free substrate surface between them offers uniform adhesion conditions for the chemically sensitive coating. Adelayline needs automatic gain control and matching to demonstrate tolerable insertion attenuation.

The resonators in contrast have small attenuation than most delay lines and do not need any matching. The oscillator design is simple as compared with delay line in terms of phase change. In this paper, we report the fabrication and performance of a SAW resonator operating at center frequency of 500 MHz for detection of Sarin (through dimethyl methylphosphonate-DMPP).

2. RESONATOR DESIGN AND LAYOUT

The resonator is based on standing surface waves. These can be configured to act as narrow band resonant circuits by arranging for incident and reflected travelling wave components to interfere with each other is a coherent manner. Fig. 1 shows a schematic layout of a two port SAW resonator. In operation, surface waves emitted from both sides of excited IDT are constructively reflected at center

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frequency by two SAW reflecting gratings, to form standing waves. These are characterized in terms of amplitude or of piezoelectric surface potential.

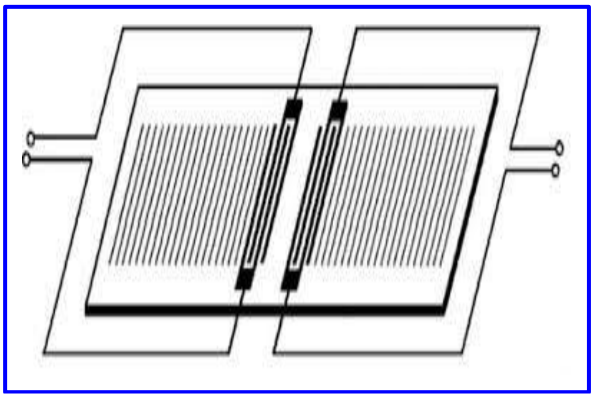

Fig. 1: Schematic diagram of a two port SAW resonator (after Maria *et al.* 2009)

The elements of SAW reflection gratings comprise of periodic discontinuities. These consist of open thin metal strips. The no. of strips required for near total reflectivity depends on reflection mechanism. In case of dominant piezoelectric shortening e.g. Lithiumniobate substrate, total reflection is achieved by few metal strips. In case of dominant mass loading e.g. ST-quartz substrate of, a few thousand grating elements are required to achieve a reflection coefficient close to unity. To keep device size realistic, SAW resonator designs on quartz substrates are normally used for frequencies above 100 MHz.

Reflection of SAW waves from a reflection grating is maximum at center frequency, for which all of individual reflections are additive, there is an arrow frequency range over which the grating reflects SAW waves with reduced efficiency. The width of this grating stop band can be specified as the frequency range over which the grating reflection coefficient exceeds some minimum value. This specification depends on design parameters including type of substrate, type of reflection grating, no. of reflector elements and other losses in grating structure (Campbell, 1989).

A SAW resonator was designed for sensing chemical/gas as per following details. Center frequency 500 MHz, material ST-quartz, number of reflecting grating elements as 200 and width of individual grating element as 1.69 micron. The design was made in AutoCAD and gdsii and is shown in fig. 2 (a), (b) and (c).

As per Campbell (1989), “The design of an efficient SAW reflection grating is a necessary but not sufficient criterion for ensuring that the two-port resonator will operate properly. A second criterion of paramount importance relates to placement of each reflection grating, with respect to its adjacent IDT. Incorrect positioning of either IDT or grating can degrade the electromechanical transduction between input and/or output voltages and acoustic resonance to the point where SAW resonance action can be lost altogether”. It is therefore, very important that the mask fabricated should have exact dimensions, so that the resonator fabricated using it works properly.

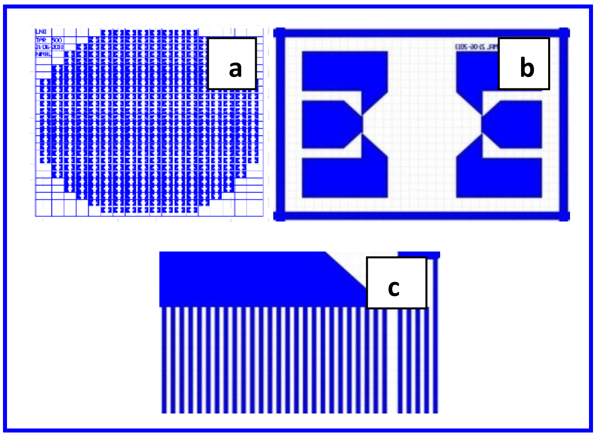

Fig. 2: Design of SAW resonator (a) Complete mask (b) One resonator and (c) Reflector elements and IDT (linewidth and spaces are 1.69 micron)

3. FABRICATION OF MASK AND SAW RESONATOR

The mask was fabricated using the data prepared in dxf (AutoCAD) and gdsii. This data was verified and fed into a LASER Pattern Generator (Heidelberg: DWL 200) after conversion and the mask was exposed using the data. Resist (AZ1518) coated pre baked master grade chrome blank of size 4”x 4” was used for the mask fabrication. After exposure, the mask plate was developed using Clariant AZ 300MIF developer. The pattern was inspected and subsequently, it was etched using Chrome etchant. Finally the photoresist was removed from mask by using stripping solution. The mask was inspected for quality and cleaned. This was then used for fabrication of SAW resonator. The final mask images are presented in fig. 3 (a), (b), (c) and (d). The critical dimension for this mask was 1.69 µ. The dimensions of fabricated structure were between 1.720 and 1.682 micron (difference from CD: 8 to 30 nm-within tolerance limit of 5 % or 0.1 µ), showing a perfect mask.

Standard photolithography (PLG) technique was utilized for device fabrication. High purity aluminum (99.999 %) was deposited on bare 3" quartz substrate using thermal evaporation system (HindHivac). The coated thickness was controlled to 2000 Å by using a thickness monitor. The substrate was then coated with AZ 1505 positive photo-resist using spin coater to a thickness of 1 μm. The coated substrate was pre-baked at 90 °C for 30 min to dry solvents and enhance adhesion in an Oven. The substrate was then exposed using i-line (365 nm) mask aligner (MJB3, Karl Suss) through photo-mask. The exposed photo-resist was then developed and examined under microscope. Post baking of the developed substrate was carried out at 90 °C to dry the developer for about 30 min prior to examination. The exposed aluminum in between the photo-resist was then etched by an electrochemical etching process. Finally the photo-resist was cleaned by Acetone. The pictures of the device are shown in fig. 4. The devices were then diced out of the wafer in set of two (for dual device configuration) using dicing machine and the diced chips were packaged. The device pads were bonded to the package pins using 1 mil aluminum wire.

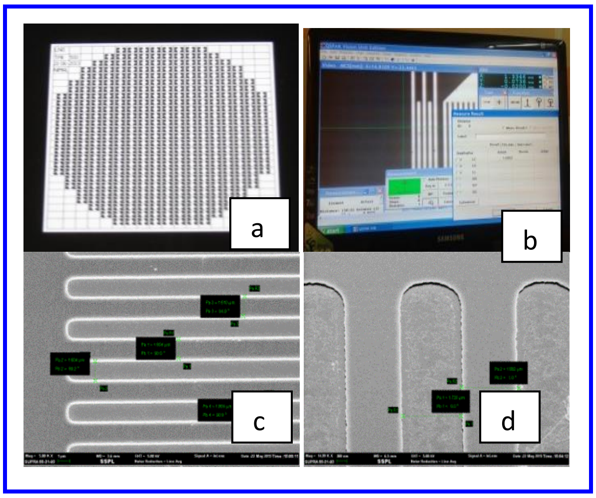

Fig. 3: Fabricated mask images (a) Complete mask (b) Resonator elements (micrograph) (c) and (d) SEM micrograph of developed and etched CDs

4. SENSOR PREPARATION AND CHARACTERIZATION

The device wascleaned using Oxygen plasma in a Plasma Asher. SAW device was then coated withBisphenol polymer by drop dry method using methanol as a solvent in a clean room environment at room temperature. The amount of material loaded was about 50 mg on the SAW path between the IDTs. The device was then baked at 100 °C for 5 hrs in nitrogen flow to evaporate remaining solvent. The device along with the package was then fitted to a sensor cell for flowing vapours in and out as described by Nimal *et al*. 2006.

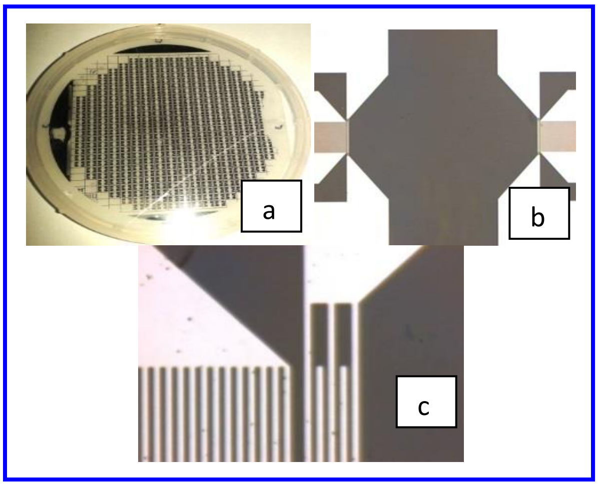

Fig. 4: Fabricated SAW resonators (a)On wafer, (b)to (c) Micrographs of CD structures

The devices were placed in the feedback loop of two stage amplifier in order to be realized as sensor oscillators. The outputs of the two oscillators, the reference and the sensor were mixed using a double balanced IC mixer and filtered by a passive low pass filter. The output was in kHz frequency which was amplified and passed to digital section for frequency measurement (Nimal *et al.* 2009). The vapor generator system for sensor characterization (developed by Singh *et al.* 2014) was used. Nitrogen was used as carrier gas and the vapor was generated by flowing nitrogen through a flask placed with a permeation vial containing DMMP (Di-Methyl Methyl Phosphonate, a simulant of Chemical Warfare Agent Sarin. The sensor was exposed alternately to carrier gas and a mixture of carrier gas and DMMP vapors.

5. RESULTS & DISCUSSION

The fabricated device was tested for its performance. First, SAW resonator was characterized for its performance and then its performance as chemical sensor was studied. The results are presented below. The fabricated SAW resonator was subjected to measurement of gain and phase in Vector Network Analyser (ZVR-Rohde and Schwarz). The resonance pattern is presented in fig 5. The acoustic resonance in SAW resonator occurs when the total phase shift of the surface wave is $\varphi=2n\pi$ within the cavity bounded by two reflection gratings. The resonance was observed at 505.7 MHz, although the device was designed to operate at 500 MHz. This is because design and fabrication of a SAW device is very complex and many factors need to be modelled/optimized. However, the resonance was observed in fabricated device, it means that the position of all elements in reflector gratings was perfect on mask (and on device). This is very important aspect of resonators, if positions of grating elements are not proper, the standing waves do not form on the surface.

The designed SAW device gas sensor was characterized using Vector Network Analyser (ZVR-Rohde and Schwarz). Fig. 6 and 7 show typical sensing curves of the prepared SAW sensor with exposure to 3 ppm DMMP vapors for two different times-short and longer durations. A frequency shift of 700 Hz was obtained, indicating sensitivity of 233 Hz/ppm.

Fischerauer *et al*. (1996) have studied the performance of a chemical sensor (SAW resonator) operating at 434 MHz. Dickert *et al.* (1998) in their study of a SAW resonator sensor for Toluene (433 MHz and 1 GHz) have inferred that limits of detectivity can be lowered by increasing the oscillation frequency of device. However, higher frequencies need thinner polymer coatings for detection. Their sensor response was 61 kHz/1000 ppm (61 Hz/ppm). Our sensor has demonstrated a frequency shift of 700 Hz for 3 ppm (about 233 Hz/ppm). Liu *et al*. (2011) have described a SAW resonator sensor for 500 MHz on ST-quartz for detection of formaldehyde. Chen *et al*. (2008) have studied a two-port SAW resonator device on ST-quartz substrates at 200 MHz for detection of mustard gas and reported sensitivity of 106 Hz/ppm. Li *et al.* (2009) have theoretically analyzed two port resonator based SAW sensor and have concluded that it is necessary to properly choose the width, the thickness and material parameters of the sensitive film in addition to the optimization of device structure, in order to improve detective sensitivity and to reduce the insertion loss.

6. CONCLUSION

In the reported work SAW resonator device operating at 500 MHz has been fabricated and studied for use as chemical sensor (Sarin-DMMP). The frequency response of the fabricated SAW device showed excellent match (insertion loss within 6 dB) as required for SAW resonator. Sensing curve of the prepared SAW sensor with exposure to 3 ppm DMMP vapors demonstrated a frequency shift of 700Hz. Bisphenol polymer coated SAW sensors can be used for sensing DMMP vapors, with proper integrated electronics.

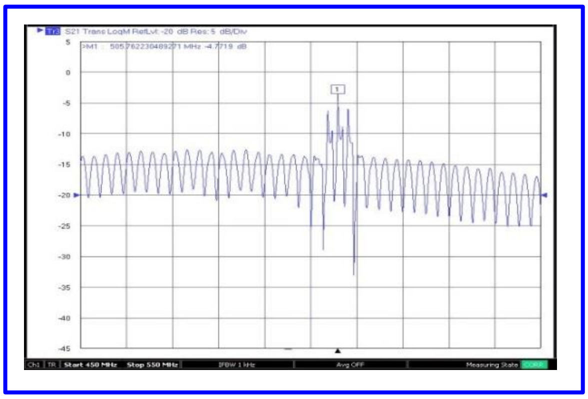

Fig. 5: Response of SAW resonator (loss 4.7 dB)

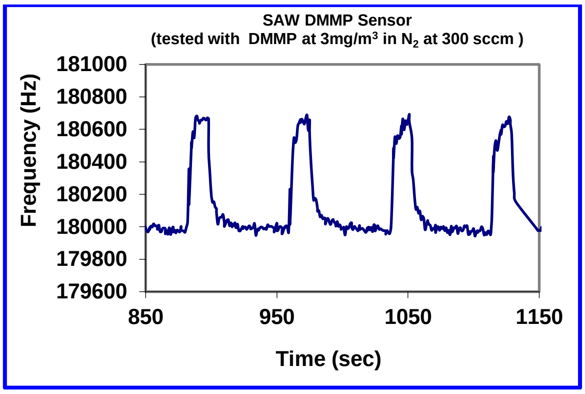

Fig. 6: Sensing curve of fabricated sensor

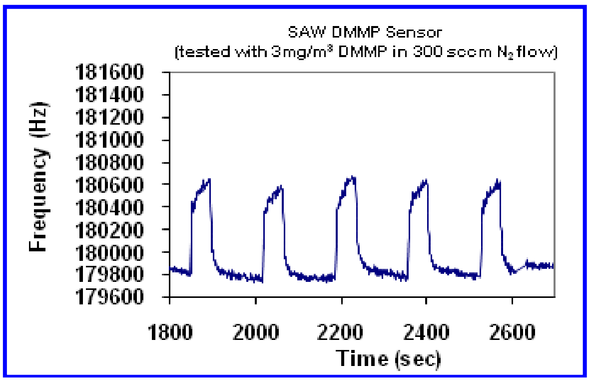

Fig. 7: Sensing curve of fabricated sensor for longertime

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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