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DEVELOPMENT OF DOUBLE SURFACE ACOUSTIC WAVE RESONATOR SYSTEM FOR HYDROGEN ND AMMONIA GAS SENSING

ZAINAB YUNUSA

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DEVELOPMENT OF DOUBLE SURFACE ACOUSTIC WAVE RESONATOR SYSTEM FOR HYDROGEN ND AMMONIA GAS SENSING

By

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

DEVELOPMENT OF DOUBLE SURFACE ACOUSTIC RESONATOR SYSTEM FOR GAS SENSING APPLICATIONS

By

ZAINAB YUNUSA

June 2015

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Acoustic wave technology has been used for gas sensing applications for several decades. A SAW device consists of a piezoelectric substrate; inter digital transducers (IDTs) and reflectors. Rayleigh waves have two types of configuration namely SAW resonator and SAW delay line. Each configuration has different structure but has similar output characteristic when employed as a sensor. The Surface Acoustic Wave (SAW) sensor has offered the development of small, lightweight, battery-free, maintenance free and multiple sensor wireless interrogation operation. Double SAW resonator (DSAWR) is a configuration that involves two SAW resonators and it has proven to be reliable in sensing applications such as temperature and strain sensor. Carbon nanotubes (CNTs) have been proven to be good sensing material with high metallic behavior. However, when they are employed as sensing materials for SAW gas sensor they cause short circuit to the IDTs. Previous works based on single SAW resonator gas sensor requires the fabrication of a guiding or protective layer which is made up of oxides so as to avoid the short circuiting of the IDTs. Based on literature reviewed, previous works have employed the DSAWR for strain and temperature sensors but it has never been deployed for gas sensing. Therefore, in this thesis, DSAWR resonator based gas sensor was developed and been deployed for gas sensing applications for the first time. The advantage of this technique is that the CNT sensor was fabricated and integrated independently which eliminates fabrication of any guiding or protective layer for the SAW resonator. Another advantage of this technique is that the same system could be used with different types of sensing layer which makes it more economical and less time consuming.

In this thesis the Double Surface Acoustic Wave Resonator System (DSAWR) for gas sensing application was proposed and developed. DSAWR system consists of two commercial SAW resonators with resonant frequencies of 433.92 and 433.42 MHz. The DSAWR system was fabricated on a PCB and deployed for gas sensing application. There are 2 types of system that were used for DSAWR gas sensing application but the systems differ in the sensing material been employed and is been configured as system 1 and system 2 sensors. System 1 sensing layer composed of functionalized multi walled carbon nanotubes with polyaniline layer which was

deployed for hydrogen sensing while system 2 sensing layer composed of polyaniline as a sensing material and was deployed for ammonia sensing.

Results obtained showed that system 2 sensor was better than system 1 sensor in terms of sensitivity. The sensitivity of system 1 sensor was found to be 3Hz/ppm at room temperature while it doubles to 6 Hz/ppm at 40 ^{0}C. System 2 sensing results obtained showed that the system has detection limit of 0.125 % with a sensitivity of 8 Hz/ppm at room temperature.

In order to investigate the sensing behavior of a new material, system 3 sensor was developed which is based on Graphene Nanoribbon (GNR) and was deployed for ammonia sensing. Results obtained showed that the novel structure could be a potential material for ammonia sensing with good sensitivity and a detection limit of 1250 ppm.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

DEVELOPMENT OF DOUBLE SURFACE ACOUSTIC RESONATOR SYSTEM FOR GAS SENSING APPLICATIONS

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Teknologi gelombang akustik telah digunakan untuk aplikasi penderiaan gas selama beberapa dekad. Peranti SAW terdiri daripada substrat piezoelektrik; transduser antara digital (IDTs) dan reflektor. Gelombang Rayleigh mempunyai dua jenis iaitu konfigurasi resonator SAW dan talian lengah SAW. Setiap konfigurasi mempunyai struktur yang berbeza tetapi mempunyai ciri-ciri output yang serupa apabila digunakan sebagai sensor.

Sensor Gelombang Akustik Permukaan (SAW) memungkinkan dalam pembangunan operasi soal siasat tanpa wayar yang berbilang sensor, kecil, ringan, tidak memerlukan bateri dan penyelenggaraan. Resonator SAW berganda (DSAWR) adalah merupakan konfigurasi yang melibatkan dua resonator SAW dan telah terbukti boleh dipercayai dalam aplikasi penderiaan seperti sensor suhu dan tekanan. Nanotube karbon (CNTs) telah terbukti menjadi bahan penderiaan yang baik dengan sifat logam yang tinggi. Walau bagaimanapun, apabila ia digunakan sebagai bahan untuk penderiaan sensor gas SAW ia menyebabkan litar pintas kepada IDTs. Penyelidikan sebelum ini yang berdasarkan sensor gas resonator SAW tunggal memerlukan fabrikasi satu lapisan pembimbing atau pelindung yang terdiri daripada oksida untuk mengelakkan litar pintas kepada IDTs.

Di dalam tesis ini sensor gas berasaskan Resonator DSAWR telah dibangunkan. Kelebihan teknik ini adalah sensor CNT telah difabrikasi dan diintegrasi secara berasingan yang menyingkirkan fabrikasi sebarang lapisan pembimbing atau perlindungan untuk resonator SAW itu. Satu lagi kelebihan teknik ini adalah bahawa sistem yang sama boleh digunakan dengan pelbagai jenis lapisan penderiaan yang menjadikan ia lebih ekonomi dan kurang memakan masa.

Dalam tesis ini, Sistem DSAWR untuk aplikasi penderiaan gas telah dicadangkan dan dibangunkan. Sistem DSAWR terdiri daripada dua resonator SAW komersial dengan frekuensi resonans 433.92 dan 433.42 MHz. Sistem DSAWR telah difabrikasi pada PCB dan digunakan untuk aplikasi penderiaan gas. Terdapat 2 jenis sistem telah digunakan untuk aplikasi penderiaan gas DSAWR namun sistem-sistem

tersebut berbeza dalam bahan penderiaan yang digunakan dan dikonfigurasikan sebagai sensor sistem 1 dan sistem 2. Lapisan penderiaan sistem 1 terdiri daripada fungsionalisasi karbon nano tiub berbilang dinding dengan lapisan polyaniline yang digunakan untuk penderiaan hidrogen manakala lapisan penderiaan sistem 2 terdiri daripada polyaniline sebagai bahan penderiaan dan digunakan untuk penderiaan ammonia.

Hasil kajian menunjukkan bahawa sensor sistem 2 adalah lebih baik daripada sensor sistem 1 dari segi sensitiviti. Kepekaan sensor sistem 1 didapati adalah 3Hz / ppm pada suhu bilik manakala ia berganda kepada 6 Hz / ppm pada 40 ^{0}C. Keputusan penderiaan Sistem 2 yang diperolehi menunjukkan bahawa sistem itu mempunyai had pengesanan 0.125 dan 8 Hz / ppm pada suhu bilik.

Dalam usaha untuk menyiasat tingkah laku penderiaan bahan baru, sensor sistem 3 telah dibangunkan yang berdasarkan GNR dan telah digunakan untuk penderiaan ammonia. Keputusan yang diperolehi menunjukkan bahawa struktur novel tersebut berpotensi untuk dijadikan bahan untuk penderiaan ammonia dengan sensitiviti yang baik dan had pengesanan 1250 ppm.

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I certify that a Thesis Examination Committee has met on 10 June 2015 to conduct the final examination of Zainab Yunusa on her thesis entitled "Development of Double Surface Acoustic Wave Resonator System for Hydrogen and Ammonia Gas Sensing" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS

SAW	Surface Acoustic Wave
DSAWR	Double Surface Acoustic Wave Resonator
IDT	Inter digital transducers
VNA	Vector Network Analyzer
CNT	Carbon nanotubes
SWNT	Single walled nanotubes
MWNT	Multi Walled Nanotubes
CVD	Chemical Vapour Deposition
CCVD	Catalytic Chemical Vapour Deposition
ACCVD	Alcohol Catalytic Chemical Vapor Deposition
GNR	Graphene nanoribbons
PCB	Printed circuit board
SEM	Scanning electron microscope
TEM	Transmission electron microscope
HRTEM	High Resolution Transmission Electron microscope
EDX	Energy Dispersive X-ray
AFM	Atomic force microscopy
XRD	X-Ray diffraction
Pani	Polyaniline
R	Resistance
f	frequency

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

A gas sensor is a device that detects the presence of gases in the environment. The ever increasing demand for developing gas sensors becomes imperative due to its vast application which ranges from environmental and air-quality monitoring, automotive and industrial control. In view of some legislative initiatives which are aimed at pollution control and human exposure to hazardous gases, development of portable and hand-held gas sensing systems becomes rather critical. There are different types of gas sensor technologies which have been used for several decades.

In the earlier centuries, traditional gas detection technologies were developed whereby the system sounds an audio alarm in order to notify people when there is a gas leakage from a harmful or poisonous source. This method is not very reliable because it is required to obtain accurate real-time measurements of the concentration of a target gas. For many centuries, different gas sensor technologies have been used for gases detection including semiconductor gas sensors, catalytic gas sensors, electrochemical gas sensors, optical gas sensors and acoustic gas sensors.

An ideal sensor is characterized based on some performance indicators such as high sensitivity, good selectivity, high detection limit, good repeatability and reproducibility, high response time and recovery time, compactness and low cost. All the gas sensors technologies listed above provides the aforementioned characteristics but have some limitation on the size, power consumption and the potential of using wireless capabilities.

Acoustic wave technology can offer the technology for gas detection and has been used for a variety of wireless sensor applications for some decades. The Surface Acoustic Technology (SAW) has offered the development of small, lightweight, battery-free, maintenance free and multiple sensor wireless interrogation operation (Hamidon, 2005). SAW components have been used as a filter or resonator in mobile phones in the telecommunication industry. They are also used as sensors for pressure, torque, acceleration, humidity, temperature, chemical and biological applications.

1.2 Surface Wave Acoustic Technology Gas Sensor

Surface acoustic wave technology involves the use of the SAW device in different technological applications. Surface acoustic waves were first discovered by Lord Rayleigh in 1885 and they were developed based on Rayleigh waves. A Rayleigh SAW is composed of two mechanical displacement components in the saggital plane which includes the plane containing the direction of propagation and the surface normal.

There are two types of acousto-electronic devices which are based on Rayleigh SAW sensors namely: delay-line or resonator. The frequency of operation of Rayleigh wave sensors is usually lies between 40MHz-600MHz (Dorozhkin & Rozanov, 2001) and in GHz range (Rodríguez-Madrid et al., 2012) . Both configurations have the same response behavior when employed as a sensor. However, they differ from each other in their design; a delay line has two receiving and transmitting inter digital transducer (IDT) whereas a resonator has one IDT placed at the resonator cavity. However, their mechanism of response is the same and they also have similar output characteristics. A delay line is simpler to design compared with the resonator that is why it is mainly preferred for practical applications.

A resonator and a delay line could be either single or two-port. A single-port delay line consists of a propagation path between one IDT and one or more reflectors. A two-port SAW delay line consists of a propagation path between two separate IDTs, the first serves as a transmitting transducer and the second serves as a receiving transducer so as to convert the SAW back to electrical form. A single-port resonator makes use of one IDT structure in the centre between two reflectors while the two-port resonator consists of two IDT structures in between two reflectors. The function of the reflector is to reflect an incident wave completely over a narrow band of frequency and also to reduce energy loss in the system so that it can produce a narrow and stable signal.

The first acoustic gas sensor was discovered by King in 1964 (Dorozhkin & Rozanov, 2001) and was based on the measurement of bulk acoustic waves (BAW) in a piezoelectric quartz crystal resonator which is sensitive to mass changes. After intensive reVHDUFKVWXGLHVLQPLG¶FKHPLFDOVHQVRUVIRULQGXVWULDODWP pollutants were developed. Since piezoelectric quartz resonators were used, these types of sensors were called quartz microbalances (QMB). There are different types of acoustic wave sensors which are based on the type of wave propagation.

The principle of operation of acoustic chemical sensor is described as follows; when a receptor film is introduced unto the vibrating surface of a transducer that is activated by an electronic device, the characteristics of the receptor film such as its mass and thickness are changed when exposed to an analyte. This change directly affects the vibration frequency, amplitude and phase. The shift resulting from the frequency changes, amplitude, phase or velocities are further translated into analyte concentration of the target gas.

The main advantages of using SAW technology is high sensitivity, low power consumption, wireless capability and can be placed on moving or rotating parts and in hazardous environment (Drafts, 2001). The SAW device is also technologically compatible because its fabrication process is similar to that of other microelectronic devices. Single SAW resonators have been used for gas sensing and have proved to be reliable and successful (Chevallier, Scorsone, & Bergonzo, 2011a; David et al., 2012; Raj, Singh, Nimal, Sharma, & Gupta, 2013; S.-H. Wang, Kuo, & Shen, 2011). However, the SAW is a very sensitive device as any slightest drift in temperature or other environmental disturbances may cause a shift in resonance frequency. Therefore a means of compensations of this drifts in resonance frequency needs to be implemented.

In this project a Double Surface Acoustic Resonator (DSAWR) system is proposed for gas sensing. This system is anticipated to provide protection against the radio channel effect and offers temperature compensation as shown by (Binhack, Buff, Klett, Hamsch, & Ehrenpfordt, 2000; M.Binhack, S.Klett, E.Guliyev, W.Buff, M.Hamsch, 2003). It has also potentialities for wireless applications. The L-matching circuit was chosen due to its simplicity and linearity, this simplicity in terms of less number of elements will improve its sensitivity towards gas detection.

The proposed DSAWR sensor system is shown in Figure 1.1. It consists of two commercial resonators namely R03101 and R03112 (RF monolithics) each connected to its matching system and a capacitor for tuning while the resistor which is connected in parallel with the capacitor is used as the sensing layer.

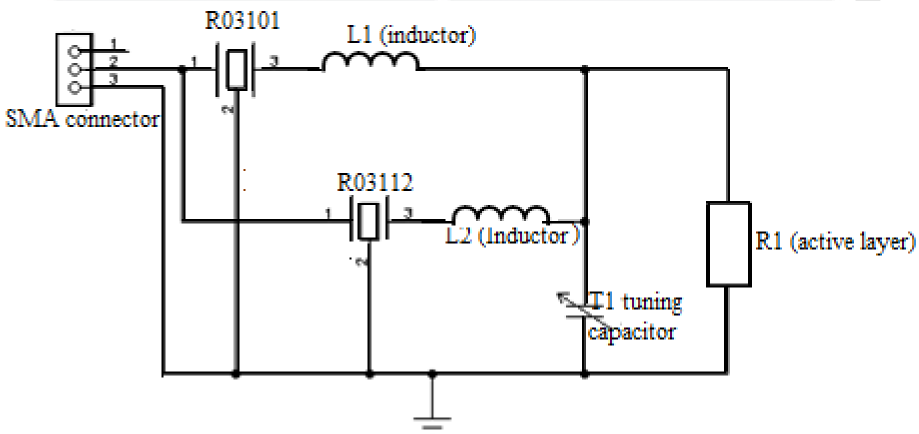

Figure 1.1 Schematic of the Proposed System on printed circuit board

1.3 Problem Statement

Nowadays, with the increased growth of worldwide industries, a lot of pollutants are injected into the environment either in air, water or land. The rate of pollutions becomes worst in developing countries such as Malaysia. With recent legislatives imposed by the government so as to harness this problem, great efforts have been made during the last decades to improve the quality of water but air pollution is not easily reduced. One of the main sources of air pollution is in power generation which is as a result of fossil fuel consumed by the power plants.

In order to reduce the use of fossil fuel consumption, the interest on using hydrogen as a clean energy source or a fuel gas has been increased remarkably because it is renewable, abundant and efficient with zero emissions. Hydrogen is also used extensively in some industries to make ammonia, methanol and rocket fuel and also including replacement of natural gas in warming home and powering hot water heaters (Winter, 2009). Therefore safety has become the first priority in using hydrogen gas as fuel like other gas fuels; hydrogen is flammable and potentially dangerous.

The explosive limit of hydrogen is more than 4% (Hübert, Boon-Brett, Black, & Banach, 2011), therefore a careful handling, storage and transportation is required. In this thesis all concentrations of hydrogen gas are expressed in percentage so as to benchmark the detection limit with the flammability limit of 4 %. However, the conversion of % to ppm is that 1 % of concentration of hydrogen gas in air is equivalent to 10,000 ppm of hydrogen gas in air. The monitoring of the concentration is very crucial so as to avoid accidents resulting from hydrogen explosions. Therefore, a reliable, sensitive and selective gas sensor is required for this job. A hydrogen gas sensor which uses a DSAWR will be developed in this project.

In addition, the proposed system will also be tested towards ammonia. This is because ammonia is colorless, poisonous and its presence in air interferes with the oxygen content. The hazards associated in both humans and animals are summarized in Tables 1.1 and 1.2. From Table 1.1 it could be seen that the minimum ammonia concentration to cause some health symptoms in humans is 25 ppm. The health symptoms complicates with increase in ammonia gas concentrations. At a concentration of 5000 ppm continuous exposure could lead to death and full protection is recommended at concentrations greater than 15, 000 ppm. At very high concentrations of greater than 150,000 ppm the gas is flammable at 50 ^{0}C.

Similarly, ammonia gas also poses serious health hazards to animals and a study was carried out on different animals (Health, 1992) in order to investigate exposure of different ammonia gas concentrations and the results are summarized and presented in Table 1.2. Based on the studies, it was observed that exposure of 5000-6000 ppm of ammonia gas concentrations causes some health indications and the concentration of 9900 ppm in cats and rabbits. Similarly, exposure of 2000 ppm of ammonia will cause harmful effect in rats while for rabbits and dogs limit of exposure that causes health hazards was observed to be 658 ppm for several weeks.

Therefore with the toxicity of ammonia gas and its health implications on both humans and animals, a very reliable gas sensor is required to detect low traces of concentrations of ammonia in air.

One of the suggested forms for the detection of these dangerous gases is the use of Surface Acoustic Wave (SAW) sensors. This is due to their small size, high frequency and sensitivity, low cost in mass production, low power consumption, simple integration and remotely controlled (wireless). As earlier stated in section 1.2, there are two types of SAW: delay-line and resonator. Delay-line focus on the time delay or the phase of the transmitted signal to the receiving signal due to the detection of the gases while resonator focus on the resonance frequency shift. Since the SAW device is sensitive to temperature, most SAW sensors uses a single SAW resonator and small drifts in temperature changes could not be compensated. Therefore, in this project a Double SAW (DSAW) resonator system is proposed for application in gas detection so as to investigate its potential for use in wireless system.

Table 1.1 Health symptoms of ammonia in humans
(www.engineeringtoolbox.com/ammonia, n.d.)

Ammonia concentration in air (ppm)	Health symptoms
<25	This is the maximum permissible limit which could be detected by smell
30	Causes discomfort in breathing with a maximum exposure of 15 minutes
50	maximum exposure limit
100	Causes eye irritation, throat and mucous membrane which may withstand tolerance in 1-2 weeks with no adverse effects.
140	May cause moderate eye irritation with no long-term effect of exposures less than 2 hours
400	Causes moderate throat irritation and may damage the mucous membranes with more than one hour exposure
500	This limit causes an immediate danger to life
1000	Destructive to the airway
1700	Short exposures of less than 30 minutes might lead to death
5000	Causes hazard to life immediately
>15000	At this limit a full body protection is required
160,000-170,000	Flammable limit in air at 50^oC

Table 1.2 Hazards of ammonia gas to animals [14]

Animals	Ammonia concentration (ppm)	Health symptoms
Guinea pigs	5000-6000 ppm for 30 to 120 minutes	Induced blindness
Cats and rabbits	9900 ppm for 1 hour	Severe bronchiolar damage and alveolar congestion
Rats	2000 ppm for 4 hours	Interstitial pneumonitis with renal tubules
Rabbits and dogs	658 ppm for weeks	Corneal opacity and eye irritation

The system will be tested towards both hydrogen and ammonia with different sensing materials. The response of the gas will be measured as the frequency shift i.e. the difference between the resonance frequencies of the two resonators. The advantage of this system is that it is suitable for passive and wireless applications and could be used for compensating slight drifts in temperature. The L-matching circuit is used for the matching circuit because of its simplicity and linearity and improved sensitivity (M.Binhack, S.Klett, E.Guliyev, W.Buff, M.Hamsch, 2003). In this project, the resonator will be used, because we want to measure the changes in the gas concentration from the change in the resonance frequency.

In order to enable the commercial operation of the sensor, the chosen transmission medium should be available worldwide. The use the industrial, scientific and medical (ISM) band is preferable because it offers license free communication in most countries. A frequency band of 433.92 MHz center frequency will therefore be used for the SAW resonator chosen for this project.

1.4 Objectives

The ultimate goal of this research is to develop a DSAWR system capable of detecting flammable gas such as hydrogen and toxic gas such as ammonia. Previous researches based on literature usually employ a single resonator system for sensing applications. It therefore becomes interesting to explore and investigate the potential of a DSAWR as a new mechanism for gas sensing applications. In order to realize this aim, the following objectives will be embarked upon.

1. Deployment of commercial SAW resonators operating at a frequency of 433.92 MHz and 433.42 MHz for the design of the DSAWR system that consists of a matching circuit. Simulation and analytical techniques will be used to get the optimal values of an L-type matching circuit configuration that consists of an inductor and a capacitor. Finally the whole system will be fabricated as shown in the schematic of the PCB as shown in Figure1.1.

2. Application of DSAWR system for gas detection.
   The developed DSAWR system will be employed for gas sensing applications towards different concentrations of hydrogen from 0.5-2% and ammonia from 0.125-1% which is equivalent to 1250-10,000 ppm with reference to Table 1.1 this range of concentration causes immediate hazards to humans which may lead to death.

3. Fabrication of the active layer for the sensor
   For this stage, Chemical Vapor Deposition (CVD) method will be used to fabricate different types of carbon nanostructures as the sensing layer. The carbon nanostructure will be directly synthesized on a piezoelectric substrate and then characterized. The locally synthesized carbon nanostructures will also be investigated for gas sensing applications.

The methodology for the overall design of the project is depicted in a block diagram as shown in Figure 1.2. This involves different stages to accomplish the objectives of the research work.

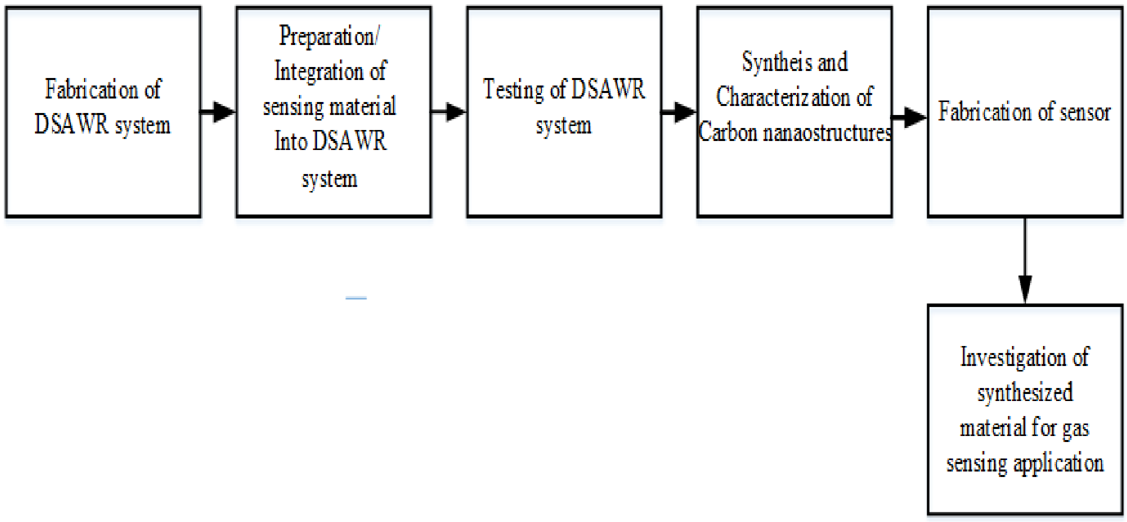

Figure 1.2 Block diagram of the overall research methodology

1.5 Scope and Limitation

The substrate that was used is gallium orthophosphate ($GaPO_4$) which is piezoelectric in nature. It was selected because of its high thermal stability and high electromechanical coupling. The high thermal stability makes $GaPO_4$ the preferred choice of substrate support for the direct synthesis of carbon nanostructures using CVD. This requires a high temperature of about 800^0C so a substrate with high thermal stability is required. Based on the literature reviewed, there has been no research works involving the use of $GaPO_4$ as a substrate support for the direct synthesis of carbon nanostructures. The reason could be due to its high cost but in this research, and it was proposed to be used as a substrate for the active layer fabrication so as to have a high thermal stability during synthesis and to explore its potentiality for gas sensing application. The Alcohol Catalytic Chemical Vapor Deposition (ACCVD) will be used throughout the research work.

The proposed system which comprises of two commercial SAW resonators R03101 and R03112 (RFM Monolithics Limited) will be used with an L-matching network only and will be fabricated on PCB. The limitation of the PCB is that it could not withstand temperature higher than 120 ^oC higher and the passive components also could not withstand high temperature during measurement. This limits its application for high temperature gas sensing application and makes it only feasible for low temperature only.

This system will then be used for gas detection application by testing it towards different concentrations of hydrogen and ammonia gases respectively. The range of gas concentration supported by the available gas measuring set up is 0.5 % to 2 % for hydrogen while for ammonia gas it is 0.125 % to 1 % which is equivalent to 1250-10,000 in ppm.

For the development of the DSAWR system, fabrication and simulation will both be employed so as to investigate the system behavior and for comparison purpose. Simulation using CST design studio will be done to get the optimized values for the matching and the DSAWR with the sensing layer.

1.6 Thesis Layout

Chapter one presents the Introduction, problem statement, aims and objectives of study, scope, limitations and contribution of the thesis and novelty of the project.

Chapter two discusses about the Review on Carbon Nanostructures as Sensing materials and their applications in SAW gas sensing. It will also discuss about the local synthesis of the sensing material using Chemical Vapor Deposition Technique.

Chapter three will discuss about the review on DSAWR system for sensing application and the proposed system to be used in the study and some preliminary simulation results.

Chapter four will present the experimental set-up for thermal CVD and ACCVD for the synthesis of carbon nanostructures and the equipments for characterization of these structures. It will also present the experimental gas sensing measurement set-up for both DSAWR method and electrical based method.

Chapter five presents the results from micro-characterization of sensing materials and results from measurement of the whole DSAWR system when applied to gas detection.

Chapter six will present the results from simulation of the whole system using CST design studio suite.

Chapter seven summarizes the findings and suggestions for future work.

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