

FLEXIBLE ZINC OXIDE SURFACE ACOUSTIC HYDROGEN GAS SENSOR  
BASED ON GRAPHENE OXIDE SENSING LAYER

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## ABSTRACT

This thesis presents the design and development a flexible surface acoustic wave (SAW) gas sensor. Fabrication and characterization of SAW device, nanostructure material, and the gas sensing performance were examined. The flexibility of the SAW substrate is highly essential due to the uneven and curved surface. The investigated structure was based on three basic conditions of the device, which are flat, bend in, and bend out. Based on the conditions, the devices were tested for the electrical and gas sensing performances to hydrogen (H<sub>2</sub>) gas. The design of the flexible SAW gas sensor was completed using a simulation process prior to fabrication. The SAW propagation and properties were investigated using finite element method (FEM) simulation. It was observed that at bending inward radius of 1.5  $\mu\text{m}$ , the total displacement and frequency shift increased by 24.5% and 89%, respectively. The simulated nanostructure sensing elements have improved the sensitivity of the gas sensor by 85.5%. For the sensing element, simulation was conducted to investigate the graphene oxide effect on bending (warping) surface towards gas. From this study, a further increase of warping angle from 180° to 270° has enhanced the binding energy. The sensor was fabricated by depositing a piezoelectric layer, interdigitated electrodes, and nanostructured material. Zinc oxide (ZnO) was deposited as the piezoelectric layer using radio frequency (RF) magnetron sputtering with different parameters and characterised using atomic force microscopic (AFM), field emission scanning electron microscopy (FESEM), and x-ray diffraction (XRD). Based on the investigation of material characteristics and surface morphology of ZnO sputtered on polyimide (PI), higher RF power increased the deposition rate at 38% from 150 to 200 W, meanwhile at 300 W, the deposition rate spiked to 67%. The S21 measurement provided insertion loss (IL) and frequency response of the SAW device. The thickness of piezoelectric thin film significantly affected the frequency response and phase velocity of the acoustic wave. The measured response of graphene nanosheet flexible SAW sensor at room temperature was taken. The radii of curvature were defined as 10 mm for bend in and bend out. The frequency shift increased in the bend in condition compared to bend out and flat conditions. The graphene oxide nanosheet sensitive element conductivity increased when electron was injected into the device surface since H<sub>2</sub> is a reducing gas. Therefore, the centre frequency of the acoustic wave velocity decreases significantly when the sensor exposed to the H<sub>2</sub> gas. The SAW gas sensing performance of the investigated nanostructure materials provides a way for further investigation to future commercialisation of these types of sensors for different types of flexible substrates

## ABSTRAK

Dalam tesis ini, kajian adalah berkisar tentang penderia gas gelombang permukaan akustik (SAW) yang fleksibel. Pembikinan dan pencirian peranti SAW, bahan struktur nano serta prestasi tindak balas gas telah diperiksa. Kebolehlenturan substrat SAW sangat penting kerana permukaan yang tidak rata dan melengkung. Struktur yang dikaji adalah berdasarkan tiga keadaan asas peranti iaitu rata, membengkok masuk, dan membengkok keluar. Berdasarkan keadaan tersebut, prestasi elektrik dan tindak balas gas penderia diuji terhadap gas hydrogen ( $H_2$ ). Reka bentuk penderia SAW gas yang fleksibel dilengkapkan menggunakan proses simulasi sebelum pembikinan. Perambatan dan sifat gelombang penderia kemudian dikaji dengan menggunakan simulasi FEM. Didapati bahawa pada radius membengkok masuk sebanyak  $1.5 \mu m$ , jumlah anjakan dan perbezaan frekuensi masing-masing meningkat sebanyak 24.5% dan 89%. Elemen tindak balas struktur nano telah meningkatkan kepekaan penderia gas sebanyak 85.5%. Untuk unsur deria, simulasi dilakukan untuk menyiasat kesan grafena oksida pada lengkungan (lenturan) permukaan terhadap gas. Dari kajian ini, peningkatan lebih lanjut dalam sudut melengkung dari  $180^\circ$  hingga  $270^\circ$  telah meningkatkan tenaga pengikat. Penderia dibikin dengan mendeposit lapisan piezoelektrik, elektrod terpadu dan bahan berstruktur nano. Zink oksida (ZnO) didepositkan sebagai lapisan piezoelektrik menggunakan percikan magnetron frekuensi radio (RF) dengan parameter yang berbeza dan dicirikan menggunakan mikroskopik daya atom (AFM), mikroskop elektron pengimbas pancaran medan (FESEM) dan pembelauan sinar-x (XRD). Berdasarkan penyiasatan ciri-ciri bahan dan morfologi permukaan ZnO terhadap poliimida (PI), daya RF yang lebih tinggi meningkatkan kadar pemendapan pada 38% dari 150 hingga 200 W, manakala pada 300 W kadar pemendapan melonjak menjadi 67%. Pengukuran  $S_{21}$  memberikan kehilangan sisipan (IL) dan tindak balas frekuensi terhadap peranti SAW. Ketebalan filem piezoelektrik memberi kesan terhadap tindak balas frekuensi dan halaju fasa gelombang akustik. Tindak balas yang diukur pada suhu bilik penderia SAW fleksibel kepingan nano diambil. Jejari kelengkungan didefinisikan sebagai 10 mm untuk membengkok masuk dan membengkok keluar. Peralihan frekuensi meningkat dalam keadaan membengkok masuk berbanding keadaan membengkok keluar dan rata. Kekonduksian elemen sensitif grafena oksida meningkat apabila elektron disuntik ke permukaan peranti kerana  $H_2$  adalah gas pengurang. Oleh itu, frekuensi pusat halaju gelombang akustik menurun dengan ketara apabila penderia didedahkan dengan gas  $H_2$ . Prestasi penderia gas SAW berkaitan bahan struktur nano yang dikaji membuka ruang untuk kajian lanjut untuk pengkomersialan jenis penderia berbeza terhadap substrat kebolehlenturan berbeza..

## TABLE OF CONTENTS

	<b>TITLE</b>	<b>PAGE</b>
	<b>DECLARATION</b>	<b>iii</b>
	<b>DEDICATION</b>	<b>iv</b>
	<b>ACKNOWLEDGEMENT</b>	<b>v</b>
	<b>ABSTRACT</b>	<b>vi</b>
	<b>ABSTRAK</b>	<b>vii</b>
	<b>TABLE OF CONTENTS</b>	<b>viii</b>
	<b>LIST OF TABLES</b>	<b>xii</b>
	<b>LIST OF FIGURES</b>	<b>xiii</b>
	<b>LIST OF ABBREVIATIONS</b>	<b>xix</b>
	<b>LIST OF SYMBOLS</b>	<b>xxi</b>
	<b>LIST OF APPENDICES</b>	<b>xxiv</b>
<b>CHAPTER 1</b>	<b>INTRODUCTION</b>	<b>1</b>
	1.1 Introduction	1
	1.2 Problem Background	1
	1.3 Problem Statement	2
	1.4 Research Objectives	3
	1.5 Research Scopes	4
	1.6 Significance and Original Contribution of This Study	4
	1.7 Thesis Structure and Organization	4
<b>CHAPTER 2</b>	<b>LITERATURE REVIEW</b>	<b>7</b>
	2.1 Introduction	7
	2.2 Surface Acoustic Wave (SAW) Based Gas Sensor	7
	2.2.1 Basic Structure of Conventional Based Gas Sensor	7
	2.3 Flexible SAW Sensor	19
	2.4 Developed Flexible SAW Hydrogen Gas Sensor	25

2.4.1	Printable Material Selection	26
2.4.1.1	Substrate Selection	26
2.4.1.2	Printable Material and Substrate Compatibility	31
2.5	Gas Sensing Mechanism for Graphene Based Sensor	39
2.5.1	Flexible Graphene- Based Electronic Devices and Hydrogen Sensors	43
2.5.2	Zinc Oxide as a Piezoelectric Material	47
2.6	Summary	50
<b>CHAPTER 3</b>	<b>SIMULATION OF FLEXIBLE SAW GAS SENSOR</b>	<b>51</b>
3.1	Introduction	51
3.2	Device Structure	51
3.2.1	Interdigital Transducer (IDT)	51
3.2.2	Piezoelectric Material	54
3.2.3	Simulation Design	55
3.2.4	Modelling Techniques	57
3.3	COMSOL Multiphysics™ Finite Element Simulation	58
3.4	Flexible SAW Gas Sensor Structure	59
3.4.1	Design Parameters	60
3.4.2	Theory and Materials	61
3.5	Geometry Setting	64
3.6	Physics Settings	68
3.6.1	Subdomain and Material Settings	68
3.6.2	Boundary conditions	69
3.6.3	Mesh Generation	71
3.6.4	Performance Analysis	71
3.6.4.1	Eigenfrequency Analysis	71
3.7	Results and Analysis	72
3.7.1	Eigenfrequency Analysis	75
3.7.2	Total Displacement	76
3.7.3	Velocity	78

3.7.4	Frequency Shift	79
3.7.5	Sensitivity	81
3.8	Sensing Element Simulation	81
3.8.1	Computational Details	82
3.9	Summary	86
<b>CHAPTER 4</b>	<b>FABRICATION OF FLEXIBLE SAW HYDROGEN GAS SENSOR</b>	<b>87</b>
4.1	Introduction	87
4.2	Fabrication of SAW Hydrogen Gas Sensor	87
4.2.1	Piezoelectric Deposition by RF Magnetron Sputtering	88
4.2.2	SAW Transducer Fabrication	92
4.2.2.1	Inkjet Printing Process	92
4.2.2.2	Screen Printing Process	94
4.2.3	Sensing Element	97
4.3	Summary	99
<b>CHAPTER 5</b>	<b>CHARACTERIZATION OF FLEXIBLE SAW HYDROGEN GAS SENSOR</b>	<b>100</b>
5.1	Introduction	100
5.2	Characterization techniques	100
5.2.1	Field Emission Scanning Electron Microscopy (FESEM)	100
5.3	X-Ray Diffraction (XRD)	102
5.3.1	Atomic Force Microscopy (AFM)	104
5.4	Characterization Results and Analysis	105
5.4.1	FESEM Surface Characterization of ZnO/PI Structure and SAW Device	105
5.4.2	XRD Characterization of ZnO on PI substrate	109
5.4.3	AFM Surface Characterization of ZnO	115
5.5	Comparison of the ZnO characterization based on research work by others	117
5.6	Summary	120



<b>CHAPTER 6</b>	<b>EXPERIMENTAL GAS SENSING SYSTEM DESIGN AND RESULTS</b>	<b>121</b>
6.1	Introduction	121
6.2	SAW Measurement Techniques	121
6.3	Design and Implementation of the Amplifier	124
6.4	RF Amplifier Design and Simulation	125
6.5	Gas Sensing Measurement Setup	128
6.6	Experimental Gas Sensing Results	130
6.7	Response Time, Recovery Time and Sensitivity	131
	6.7.1 Frequency and Phase Response of Flexible SAW Gas Sensor	132
	6.7.2 Gas Sensing Results	141
6.8	Summary	147
<b>CHAPTER 7</b>	<b>CONCLUSION AND FUTURE WORKS</b>	<b>148</b>
7.1	Conclusions	149
7.2	Future Works	151
	<b>REFERENCES</b>	<b>154</b>
	<b>LIST OF PUBLICATIONS</b>	<b>176</b>

## LIST OF TABLES

<b>TABLE NO.</b>	<b>TITLE</b>	<b>PAGE</b>
Table 2.1	SAW devices for sensor applications using rigid substrate	17
Table 2.2	SAW devices for sensor applications using flexible substrate	23
Table 2.3	Performance properties polymeric substrate for flexible electronic devices	28
Table 2.4	Function and operating frequency of SAW sensor fabricated onto polyimide substrate [40]–[44]	28
Table 2.5	Comparison with other developed SAW hydrogen gas sensor	30
Table 2.6	Comparison of features and properties for screen printing and inkjet printing.	34
Table 2.7	Comparison of features and properties for screen and inkjet printing	38
Table 2.8	The advantages and limitations of graphene-based sensing element	41
Table 3.1	SAW properties of piezoelectric material	55
Table 3.2	Flexible SAW gas sensor parameter	61
Table 3.3	Material properties for aluminum and graphene	69
Table 3.4	Boundary condition applied summary	70
Table 3.5	Comparison for optimum performance for thin film and nanostructured sensing element	81
Table 4.1	Sputtering parameters for ZnO on PI substrate	91
Table 4.2	IDT parameter used in this research	92
Table 5.1	The dislocation density, $d$ -spacing values and $c$ -axis lattice constant at (002) peak for different RF Power	113
Table 5.2	Comparison of the ZnO characterization based on other research work in terms of $c$ -axis lattice, $d$ -spacing, crystallite size, FWHMA, $2\theta$ and stress	118
Table 6.1	Oscillation frequency before and after gas and frequency shift for flexible SAW sensor 1 and 2	146

## LIST OF FIGURES

<b>FIGURE NO.</b>	<b>TITLE</b>	<b>PAGE</b>
Figure 2.1	Sensor operating principle	7
Figure 2.2	Surface Acoustic Wave gas sensor basic structure	8
Figure 2.3	The Rayleigh surface acoustic wave excited by IDT	9
Figure 2.4	The generating of sinusoidal wave on the surface of substrate	9
Figure 2.5	Classifications of SAW sensor substrates	11
Figure 2.6	Dual delay line LiNbO <sub>3</sub> SAW device with PbPc film on leftwave path	13
Figure 2.8	Development of flexible electronics in various applications	19
Figure 2.9	Mechanical characteristics for flexibility and the examples	19
Figure 2.10	The schematic diagram and the fabricated flexible surface acoustic wave device	20
Figure 2.11	The structure of 2-port SAW resonator on polymer substrate and The SEM micrograph of AlN thin films on polymer substrate	21
Figure 2.12	The flexible thin film ZnO/polyimide SAW devices. (a) Three-dimensional schematic of the developed SAW device on a ZnO/polymersubstrate; (b) Microscope image of a SAW device with 20 pairs of interdigitated transducers (IDT) figure; (c) & (d) Photographs of semi-transparent and flexible SAW devices on polyimide [34]	22
Figure 2.13	Flexible SAW gas sensor structure	25
Figure 2.14	Flexible substrate criteria	27
Figure 2.15	Ink properties on polymer substrate	31
Figure 2.16	The classification of common printing method.	32
Figure 2.17	Screen printing technique	35
Figure 2.18	Piezo inkjet printing	36
Figure 2.19	(a) Graphene honeycomb lattice of carbon atoms (b) The shape of Brillouin zone	39

Figure 2.20	Graphene forms [60]	40
Figure 2.21	The graphene oxide molecular structure	42
Figure 2.22	H <sub>2</sub> on Graphene: The binding energy and distance of H <sub>2</sub> above graphene surface [83].	45
Figure 2.23	Representation of ZnO crystal structure	48
Figure 2.24	Schematic representation of a wurtzite ZnO structure [101]	48
Figure 3.1	IDT Structure with physical dimension [107]	52
Figure 3.2	Operation of SAW device [107]	53
Figure 3.3	Reduction of 3D to 2D model [110]	57
Figure 3.4	Periodic structure in a SAW device	58
Figure 3.5	Simulation steps in COMSOL Multiphysics™	59
Figure 3.6	SAW gas sensor structure	60
Figure 3.7	2D Geometry setting for SAW gas sensor	60
Figure 3.8	Flat, Bend in and bend out which represent device bending with degree (h)	61
Figure 3.9	COMSOL Multiphysics version 3.5 model navigator	65
Figure 3.10	Geometry model for flat SAW gas sensor for graphene thin film	65
Figure 3.11	Geometry model for bend in configuration SAW gas sensor for graphene thin film	66
Figure 3.12	Geometry model for bend out configuration SAW gas sensor for graphene thin film	66
Figure 3.13	Geometry model for flat configuration SAW gas sensor for nanostructured graphene	66
Figure 3.14	Geometry model for bend in configuration SAW gas sensor for nanostructured graphene (zoomed at the middle and edge)	67
Figure 3.15	Geometry model for bend out configuration SAW gas sensor for nanostructured graphene	67
Figure 3.16	Boundary conditions for SAW sensor	69
Figure 3.17	The meshed geometry	71
Figure 3.18	Deformed displacement of SAW gas sensor	72

Figure 3.19	The deformed shape plot for propagating SAW for flat condition with graphene thin film sensing element	73
Figure 3.20	The deformed shape plot for propagating SAW for flat condition with graphene nanostructure sensing element	73
Figure 3.21	The deformed shape plot for propagating SAW for bend in condition with graphene thin film sensing element	74
Figure 3.22	The deformed shape plot for propagating SAW for bend in condition with graphene nanostructure sensing element	74
Figure 3.23	The deformed shape plot for propagating SAW for bend out condition with graphene thin film sensing element	74
Figure 3.24	The deformed shape plot for propagating SAW for bend out condition with graphene nanostructure sensing element	75
Figure 3.25	Bending radius versus operating frequency for graphene thin film as sensing element	76
Figure 3.26	Bending radius versus operating frequency for graphene nanostructure as sensing element	76
Figure 3.27	Bending radius versus total displacement for graphene thin film as sensing element	77
Figure 3.28	Bending radius versus total displacement for graphene nanostructure as sensing element	78
Figure 3.29	Bending radius versus velocity for graphene thin film as sensing element	79
Figure 3.30	Bending radius versus velocity for graphene nanostructure as sensing element	79
Figure 3.31	Bending radius versus frequency shift for graphene thin film as sensing element	80
Figure 3.32	Bending radius versus frequency shift for graphene nanostructure as sensing element	80
Figure 3.33	Graphene warped inward 90°	84
Figure 3.34	Graphene warped inward 180°	85
Figure 3.35	Graphene warped inward 270°	85
Figure 3.36	Binding energy based on warping angle inward and outward	85
Figure 3.37	Charge transfer based on warping angle inward and outward	86
Figure 4.1	Flexible SAW gas sensor fabrication process	88

Figure 4.2	Process occurred inside the vacuum chamber during sputtering process	89
Figure 4.3	Clean booth class 1000 that provides RF Magnetron Sputtering machine and vacuum chamber at IIUM	90
Figure 4.4	RF magnetron sputtering process flow for deposition of ZnO	90
Figure 4.5	Inkjet printer in the laboratory and the Inkjet drop and analysis system software (IJDAS 300)	93
Figure 4.6	Inkjet printing of silver ink on Polyimide (PI) substrate.	94
Figure 4.7	SAW device design with dimensions for mask	95
Figure 4.8	Stencil plate making process	95
Figure 4.9	Conductivity test for four samples	96
Figure 4.10	Screen printing process flow	97
Figure 4.11	Ultrasonic bath for graphene and the diluted graphene oxide	98
Figure 4.12	Drop casting process flow	99
Figure 5.1	Schematic diagram of a FESEM setup	102
Figure 5.2	FESEM laboratory	102
Figure 5.3	The reflection of an X-ray beam by lattice planes in crystal	103
Figure 5.4	Advanced X-ray diffraction laboratory	103
Figure 5.5	Laser beam deflection for atomic force microscopes	104
Figure 5.6	Force distance curve for AFM	105
Figure 5.7	Advanced Optical Microscope & Nano Raman Photoluminescence Laboratory	105
Figure 5.8	(a) FESEM micrographs of ZnO thin film deposited on PI substrate. (b) The c-axis growth oriented perpendicular to the substrate's surface.	106
Figure 5.9	The cross section of ZnO thin film deposited on PI with different thickness and RF power	106
Figure 5.10	Deposition rate vs RF power	107
Figure 5.11	Top view of ZnO sputtered on PI with different RF sputtering power	108
Figure 5.12	Grain size and ZnO thickness versus RF sputtering power	108

Figure 5.13	(a) FESEM top view of interdigitated electrodes (IDT) (b) The drop casted graphene oxide on SAW device	109
Figure 5.14	X-Ray Diffraction $2\theta$ scan of ZnO on PI substrates for different RF power (b) $2\theta$ , intensity and FWHM measurement for different RF power	110
Figure 5.15	(a) Peak position of ZnO (002) at $2\theta$ in degrees versus RF Power and (b) (FWHM) values and crystallite size versus the RF power	111
Figure 5.16	(a) $c$ -axis lattice constant and the corresponding compressive stress at different power and (b) FWHM and crystallite size vs various RF power	114
Figure 5.17	AFM images for (a) 0.367 $\mu\text{m}$ (b) 0.506 $\mu\text{m}$ and (c) 0.843 $\mu\text{m}$ of ZnO thicknesses	117
Figure 6.1	SAW measurement techniques	121
Figure 6.2	Experimental setup employs network analyzer to measure SAW phase velocity and attenuation.	122
Figure 6.3	Experimental setup for SAW phase velocity measurement using by implementing oscillator and frequency counter.	123
Figure 6.4	Three stages RF amplifier schematic	126
Figure 6.5	Comparison of simulated RF amplifier	126
Figure 6.6	Three stages RF amplifier circuit on breadboard	127
Figure 6.7	Output for the three stages RF amplifier	127
Figure 6.8	Design of PCB circuit and the experimental testing for the output of RF amplifier	128
Figure 6.9	Gas testing chamber for SAW gas sensor	129
Figure 6.10	Design of gas chamber using solidwork	130
Figure 6.11	Experimental setup for flexible SAW gas sensor system	130
Figure 6.12	Response curve	131
Figure 6.13	Measurement setup for S11 and S21 using vector network analyser	133
Figure 6.14	Transmission spectrum of flexible ZnO/Polyimide SAW device 1 for Bend in A, Bend In B, Flat and Bend Out A.	135
Figure 6.15	The frequency response versus the bending condition	136
Figure 6.16	Transmission spectrum of flexible ZnO/Polyimide SAW device 2 for Bend in A, Bend in B (c) Flat, Bend out A and Bend out B	138

Figure 6.17	(a) Frequency response versus insertion loss for SAW device 2 and (b) The Frequency response versus the bending conditions	138
Figure 6.19	Illustration of wavelength IDTs increase during bent out.	139
Figure 6.20	Full experiment for gas sensing setup	142
Figure 6.21	Oscillation frequency for SAW sensor in Bend in, Bend Out and	145
Figure 6.22	Frequency shift for flexible SAW sensor 1 and 2	146



## LIST OF ABBREVIATIONS

AFM	-	Atomic Force Microscope
AGNR	-	Graphene Nanoribbon
Al	-	Aluminum
AlN	-	Aluminum Nitride
Ar	-	Argon
AZO	-	Aluminum Doped Zinc
C	-	Carbon
CMOS	-	Complementary Metal-Oxide-Semiconductor
CO <sub>2</sub>	-	Carbon Dioxide
CSA	-	Camphor Sulfonic Acid
DFT	-	Density Functional Theory
DFT	-	Density Functional Theory
DI	-	Dionized
DOF	-	Degree of Freedom
EDA	-	Electronic Design Automation
FEM	-	Finite Element Method
FESEM	-	Field Emission Scanning Electron Microscopy
FWHM	-	Full Width at Half Maximum
GaPO <sub>4</sub>	-	Gallium Phosphate
GO	-	Grapheme Oxide
H <sub>2</sub>	-	Hydrogen
H <sub>2</sub> S	-	Hydrogen Sulphide
IDT	-	Interdigitated Transducer
IL	-	Insertion Loss
IoT	-	Internet of Things
ISS	-	Impedance Standard Substrate
LCD	-	Liquid Crystal Display
LGS	-	Langasite
LiNbO <sub>3</sub>	-	Lithium Niobate

LiTaO <sub>3</sub>	- Lithium Tantalate
MEMS	- Microelectromechanical Systems
Mo	- Molybdenum
MWCNT-	MultiWalled Carbon Nanotubes
NEGF	- Non-Equilibrium Green's Function
NH <sub>3</sub>	- Ammonia
NO	- Nitrogen Oxide
O <sub>2</sub>	- Oxygen
PANI	- Polyaniline
PCB	- Printed Circuit Board
PI	- Polyimide
PNVP	- Poly-N-vinylpyrrolidone
PVDF	- Polyvinylidene Difluoride
PZT	- Lead Zirconate Titanate
RF	- Radio Frequency
RH	- Relative Humidity
RMS	- Root Mean Square
SAW	- Surface Acoustic Wave
Si	- Silicon
SO <sub>2</sub>	- Sulphur Dioxide
VNA	- Vector Network Analyzer
XRD	- X-Ray Diffraction
ZnO	- Zinc Oxide

## LIST OF SYMBOLS

$f$	-	Frequency
$v$	-	Acoustic wave velocity
$\lambda$	-	Acoustic wave wavelength
$k^2$	-	Electromechanical coupling coefficient
$C_s$	-	Dielectric constant
$y_0$	-	Characteristic admittance of the SAW transmission line
$d$	-	Thickness
$r$	-	Cylindrically bend to radius
$a_{1,2}$	-	Triangular lattice unit vector
$b_{1,2}$	-	Reciprocal triangular lattice
$E_k$	-	Tight binding structure of graphene
$E_F$	-	Fermi energy
$K'$	-	Dirac point
$t$	-	nearest neighbour hoping integral
$h$	-	Bending radius
$T$	-	Stress tensor
$c^E$	-	Elasticity matrix
$S$	-	Strain tensor
$e$	-	Piezoelectric coupling constants
$E_k$	-	Electric field intensity
$W_e$	-	Width of electrodes
$W_{sp}$	-	Space between each electrode
$f_0$	-	Operating frequency
$v_0$	-	Velocity of wave propagation
$\rho$	-	Partial density
$P$	-	Pressure
$R$	-	Gas constant
$T$	-	Air temperature

$\Delta f$	-	Frequency shift
$U_L$	-	Left displacement
$U_R$	-	Right displacement
$\varphi_L$	-	Left boundary potential
$\varphi_R$	-	Right boundary potential
$Ea$	-	Binding energy
$E_{(Graphene + Gas)}$	-	Total energy of optimized graphene and gas molecule
$E_{(Graphene)}$	-	Total energy of optimized graphene
$E_{(Gas)}$	-	Total energy of optimized gas molecule
$N$	-	Charge transfer
$D_{ij}$	-	Density matrix
$S_{ij}$	-	Overlap matrix
$P_3$	-	$c$ -axis direction of polarization
$e_{33}$	-	Wurtzite piezoelectric stress coefficient
$e_{31}$	-	Wurtzite piezoelectric stress coefficient
$\varepsilon_{1,2}$	-	Lattice constant on $a$ -plane
$\varepsilon_3$	-	Lattice constant from 002 plane
$c$	-	Lattice constant obtained from (002) plane
$c_0$	-	Lattice constant obtained from (002) plane
$a$	-	The lattice constant along $a$ plane
$a_0$	-	The lattice constant along $a$ plane
$e$	-	Piezoelectric constant
$\varepsilon$	-	Dielectric constant along the acoustic wave propagation
$c_s$	-	Effective elastic stiffness
$v_s$	-	The sound velocity for ZnO along $c$ -axis
$F$	-	Force
$k$	-	Stiffness of the lever
$D$	-	Crystalline size
$X_{rad}$	-	FWHM of the (002) plane peak in radians
$\delta$	-	Dislocation density
$d_{hkl}$	-	$d$ -spacing
$c$	-	$c$ -axis lattice constant
$\sigma$	-	Biaxial strain
$\varepsilon$	-	Film strain along (002) orientation

$c_{film}$	-	The lattice constants obtained from (002) plane
$c_{ZnO}$	-	Lattice constant of ideal ZnO
$I_{(bkg)}$	-	Background intensity
$I_{(002)}$	-	Intensity of the peak at plane of (002)
$\varphi_A$	-	The phase shift produced by the amplifier
$\varphi_c$	-	Phase shift produced by gas chamber electrical connections
$T$	-	Signal delay
$\varphi_e$	-	Constant if the amplifier is working at stable conditions
$S$	-	Sensitivity
$R_{air}$	-	Resistance of the sensor in air
$R_{gas}$	-	Resistance of the sensor in presence of gas

## LIST OF APPENDICES

<b>APPENDIX</b>	<b>TITLE</b>	<b>PAGE</b>
Appendix A	Technical properties for silver paste	172
Appendix B	Data sheet for Graphene Oxide paste	174

# CHAPTER 1

## INTRODUCTION

### 1.1 Introduction

The purpose of this chapter is to provide a general overview and introduction for the research presented in this PhD thesis. This chapter addresses the research motivations, problem statement, objectives, scopes and the significance to knowledge.

### 1.2 Problem Background

One of the most significant sources of air pollution is electricity generation, which is caused by the fossil fuels used by power plants. Since hydrogen ( $H_2$ ) is renewable, plentiful, and reliable, as well as having zero emissions, interest in using it as a clean energy source or a fuel gas has risen dramatically in order to reduce fossil fuel usage.  $H_2$  is also widely used in a variety of industries for instance industries to make ammonia, methanol and rocket fuel and also as a replacement for natural gas in warming homes and powering hot water heaters.

However, the explosive nature of  $H_2$  gas above 4% concentration makes it highly dangerous to store, transport and use [1]. Further, the small size gas molecules of  $H_2$  are prone to leak through the smallest possible holes and cracks. Hence, the detection of  $H_2$  gas becomes essential even at trace levels.

Gas sensors are applied for facilitating the safe use of  $H_2$  in, for example, fuel cell and  $H_2$  fueled vehicles. New sensor developments, aimed at meeting the increasingly stringent performance requirements in emerging applications are presented.

Flexible and wearable sensor application potential has been great field of interest for the past several decades. The development of flexible gas sensing systems is raising a high interest among the scientific community due to their potential applications in wear-able and portable electronic products, in RFID tags. Moreover, the techniques used in the flexible gas sensing industry, such as screen and inkjet printing, enable the large-scale fabrication of low-cost effective systems [2]. Many reports have been published regarding the growth of gas sensor market.

### **1.3 Problem Statement**

There are various methods of gas detection types can be found in several papers. In the past 20 years, there was vast development of Surface Acoustic Wave (SAW) as a sensor with numerous applications ranging from very basic home appliances, advanced medical devices, automotive industry to space vehicles [3]–[5].

H<sub>2</sub> gas is used as reducing agent and as a carrier gas in the process of manufacturing semiconductors. It has been increasingly known as a clean source of energy or a fuel gas. Based on [6] leaking of hydrogen gas must be avoided as it will lead to explosion if mixed with air in ratio of 4.65-93.9 vol.%. Therefore, fast response and accurate hydrogen detector before the explosive concentration and room temperature still a great problem.

SAW gas sensors are very attractive based on their excellent sensitivity due to changes of boundary conditions for propagating acoustic Rayleigh waves. Change in physical and chemical properties can be easily detected as long as the thickness of sensitive layer is less than the wavelength of the surface wave.

Most of the SAW sensor are made on rigid substrates are not suitable for curved surface which are essential for flexible sensing devices. In 2005, before the flexible SAW sensors were proven to be utilized as temperature and humidity sensor, Preethichandra et. al [7], [8] have shown that flexible SAW sensor has an ability to measure bending curvature. Preethichandra et. al have fabricated SAW sensor on a flexible Polyvinylidene Difluoride (PVDF) substrate in order to obtain bending



curvature which will be use in a high-accuracy tele-operational robotic hand. They found that the output voltage of the SAW sensor is proportional to the curvature. Based on this ability, they suggests the possibility of devising a dynamic surface profile sensor in which has a lot of scope in biomedical applications.

Moreover, studies investigated by Tseng et al. and Ad Park et al. [9], [10] show the effect of bending on the electrical and optical characteristics of ZnO thin film. The result shows the durability of the thin film on flexible polymer produces good electrical stability and resistivity changes gradually depends on bending radius. However, there are not many research found for flexible SAW gas sensor due to difficulties in achieving high quality of piezoelectric thin film.

This is due dimension of a flexible substrate with various surface adhesion which possess low surface energies, this will cause difficulties in achieving the growth of high quality piezoelectric thin film. Most critical part is when the fabricate of flexible SAW devices are it is challenging to obtain high c-axis oriented, low surface roughness piezoelectric films with a good piezoelectric constant and this may cause by several factors. An effective approach of manufacture flexible SAW is lack causes complications in exploitation of flexible devices. Therefore, the main goal of this research is to fabricate sensor with improved quality of piezoelectric thin film.

#### **1.4 Research Objectives**

The objectives of this research are:

1. To study the propagation and analyse the properties of SAW gas sensor with graphene thin film and nano-structure sensing element via simulation using Finite Element Method.
2. To investigate the material characteristics and surface morphology of ZnO thin film that sputtered on polyimide with different sputtering parameters.
3. To investigate in detail the basic behaviour (such as electrical performance, reflection ( $S_{11}$ ) and transmission ( $S_{21}$ ) of SAW device on flexible substrate.
4. To examine the effect of bending towards the performance of SAW gas sensor.

## **1.5 Research Scopes**

In this project, a flexible Surface Acoustic Wave hydrogen gas sensor is fabricated. The design of flexible SAW gas sensor completed using simulation process prior to fabrication via COMSOL Multiphysics. Based on simulation, there are several analyses which include eigenfrequency analysis, total displacement velocity and frequency shift. The analysis is important to relate performance with the fabricated sensor. Next, the fabrication of the sensor realized by depositing piezoelectric layer using RF sputtering. Interdigitated electrodes for the sensor were deposited using print screen technique. While sensing material realized by drop casting method. All of the fabricated materials have been characterized the morphology, crystallography, orientation and film thickness using based on XRD, AFM and FESEM to observe the quality and performance. The nanostructured material deposited onto the active area of SAW device to increase the volume to surface ratio, subsequently will improve the sensor's sensitivity. The flexibility of the SAW substrate is highly essential due to the uneven and curved surface. Experimental investigation and data evaluation will be carried out to proof the ability a flexible SAW sensor for hydrogen gas sensing performance.

## **1.6 Significance and Original Contribution of This Study**

This study significantly contributes to the optimizing the growth of zinc oxide (ZnO) and its role as a piezoelectric on the flexible substrate. It also to study about the deposition morphology, crystallography, orientation and film thickness of ZnO effect on the SAW transmission characteristics. Furthermore, implementing the print screen method for IDT and effect of bending the SAW gas sensor.

## **1.7 Thesis Structure and Organization**

The thesis is primarily devoted to this topic and is divided as follow:

- Chapter 2 presents the literature review on flexible SAW gas sensor, operating principles and mechanisms. This chapter also includes the

past studies on rigid and flexible sensors.

- Chapter 3 discusses in detail the simulation of flexible SAW gas sensor using COMSOL Multiphysics to provide the preliminary results on the sensor functionality.
- Chapter 4 explains the fabrication steps for flexible SAW gas sensor which involving the deposition of piezoelectric thin film, the metallization layer deposition and the implementation of the sensing element.
- Chapter 5 characterizes the flexible SAW gas sensor piezoelectric thin film, metallization and sensing element by employing X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM).
- Chapter 6 focuses on the experimental gas sensing system design which presents the testing of the flexible SAW gas sensor on bending position and measuring the response of the sensor toward the gas.
- Chapter 7 concludes the project work based on the results drawn and future works that may be applied.

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## LIST OF PUBLICATIONS

### Journal

1. Fatini, S., Rashidah, A., Zaharah, J., Aizat, A. M. I., and Mahyuddin, A. (2017). Performance Enhancement by Implementation of Nano structure Sensing Element for Bendable SAW Gas Sensor: Simulation. *International Journal of Applied Engineering Research (Scopus)* (online special issue) (ISSN: 0973-4562).

### Conference Proceeding

1. **Fatini, S.**, Rashidah, A., Rafidah, I., Aizzat, A. M. I., Zaharah, J., and Leow, P. L. (2017). A Comparative Study on Simulation Performances of Rigid and Bendable SAW for Gas Sensor. In *AIP Conference Proceedings* 1808, 020048 (2017); doi: 10.1063/1.4975281.

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