SURFACE ACOUSTIC WAVE GAS SENSOR WITH NANOSTRUCTURED SILICON SENSING ELEMENT

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ABSTRACT

Surface acoustic wave (SAW) sensors have significant potential to monitor toxic gases, owing to their advantageous features such as fast response time, high stability, and remarkable sensitivity. Over the past few years, research has been focused on optimizing the design and fabrication of SAW sensors by adopting various materials as a sensing layer for enhanced sensitivity. Among the high gas sensitive materials available, zinc oxide (ZnO), palladium (Pd), tungsten oxide (WO_3), Gallium nitride (GaN), and indium oxide (In_2O_3) have been widely adopted. However, the high gas recovery time with regard to its response time and poor detection stability make these materials adverse for gas sensing applications. This work presents a novel microelectromechanical system (MEMS) SAW gas sensor that employs an amorphous Silicon nanostructure (Si-nanostructure) as the sensing material. Due to the high surface-to-volume ratio of Si-nanostructure, it provides high sensitivity by adsorbing more gas molecules. The fabrication of the Si-nanostructure was carried out by a low-cost metal-assisted chemical (MACE) process. The formation and structural properties of these Si-nanostructure were characterized by Field-emission Scanning Electron Microscopy (FESEM) and Energy Dispersive X-ray (EDX) analysis. The fabricated SAW sensor with Si-nanostructure sensing material was then tested with Carbon dioxide (CO_2) gas during the characterization process. The gas-sensing performance of the sensor was assessed in terms of its sensitivity performances, and then compared with previously reported SAW gas sensors. The developed Si nanostructure-based SAW sensor exhibits a frequency shift of 4.622 kHz at 2000 ppm, and a response and recovery time of 9 s and 9.5 s, respectively, when exposed to 700 ppm of CO_2 gas. It demonstrates higher sensitivity and better response than previously reported devices in the literature, and has promising potential to be employed as a primary sensing layer for next-generation MEMS SAW gas sensors.

ABSTRAK

Penderia gelombang akustik permukaan (SAW) mempunyai potensi yang besar untuk pemantauan gas toksik, kerana kelebihannya seperti kadar tindak balas yang pantas, kestabilan dan kepekaan yang tinggi. Sejak beberapa tahun kebelakangan ini, penyelidikan telah tertumpu pada mengoptimumkan reka bentuk dan pembikinan penderia SAW dengan menggunakan pelbagai bahan sebagai lapisan penderia untuk meningkatkan kepekaannya. Antara bahan lapisan sensitif dengan kepekaan tinggi terhadap gas adalah, zink oksida (ZnO), paladium (Pd), tungsten oksida (WO_3), Gallium nitride (GaN), dan indium oksida (In_2O_3) telah diguna pakai secara meluas. Walau bagaimanapun, ia menggambil masa yang lama untuk bertindak balas dengan gas dan ia mempunyai kestabilan pengesanan yang lemah menyebabkan bahan-bahan seperti ini kurang berkesan untuk digunakan dalam aplikasi pengesanan gas. Kajian dalam tesis ini memperlihatkan penderia gas SAW baru di dalam sistem mikroelektromekanik (MEMS) yang terdiri daripada struktur nano Silikon (Si-nanostructure) amorf sebagai bahan penderiaan gas. Oleh kerana nisbah permukaan-ke-atas isipadu Si berstruktur nano adalah tinggi, ia memberikan kepekaan yang tinggi dengan menyerapan lebih banyak molekul gas. Pembikinan Si berstruktur nano telah dijalankan dengan proses kimia bantuan logam (MACE) yang berkos rendah. Pembentukan dan sifat Si berstruktur nano ini dicirikan berdasarkan analisis Mikroskopi Elektron Pengimbasan Pancaran Medan (FESEM) dan sinar-X sebaran Tenaga (EDX). Penderia SAW terbikin dengan bahan penderiaan struktur nano Si kemudiannya telah diuji dengan gas karbon dioksida (CO_2) semasa proses pencirian. Penderia SAW berasaskan struktur nano Si yang telah dibangunkan memperlihatkan anjakan frekuensi sebanyak 4.622 kHz pada 2000 ppm, dan masa tindak balas dan pemulihan masing-masing 9 s dan 9.5 s, apabila terdedah kepada 700 ppm gas CO_2. Ia menunjukkan kepekaan yang lebih tinggi dan tindak balas yang lebih baik daripada peranti yang pernah dilaporkan sebelum, dan mempunyai potensi yang baik untuk digunakan sebagai lapisan penderiaan utama untuk penderia gas MEMS SAW generasi akan datang.

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LIST OF ABBREVIATIONS

MEMS	-	Microelectromechanical systems
Si	-	Silicon
CO_2	-	Carbon Dioxide
NH_4	-	Ammonia
CO	-	Carbon Monoxide
MOS	-	Metal oxide semiconductor
ZNO	-	Zinc oxide
VOC	-	Volatile organic compound
SAW	-	Surface acoustic wave
GC	-	Gas chromatography
TiO_2	-	Titanium dioxide
Fe_3O_2	-	Ferric oxide
SnO_2	-	Tin (IV) oxide
PhC	-	Photonic Crystal Cavity
QCM	-	Quartz Crystal Microbalance
CH_3OH	-	Methanol
CH_2O	-	Formaldehyde
NO_2	-	Nitrogen dioxide
H_2	-	Hydrogen
FESEM	-	Field emission scanning electron microscope
FEA	-	Finite element analysis
EDX	-	Energy dispersive x-ray
IDT	-	Interdigital transducers
ppm	-	Part per million
MgF_2	-	Magnesium fluoride
C_6H1_4	-	Hexane
SOI	-	Silicon-on-insulator
PZT	-	Lead zirconate titanate
Pb	-	Lead
PBG	-	Photonic bandgap

CdS	-	Cadmium sulphide
ITO	-	Indium tin oxide
$LiNbO_3$	-	Lithium niobate
PECVD	-	Plasma-enhanced chemical vapor deposition
MACE	-	Metal-assisted chemical etching

LIST OF SYMBOLS

s	-	Second
m	-	Diffraction order
λ	-	Wavelength
v	-	Velocity
Δf	-	Resonance frequency shift
fo	-	Fundamental frequency
Δm	-	Mass change
n	-	effective refractive index
d	-	Distance
θ	-	Angle
f_0	-	Undisturbed frequency
Vo	-	Undisturbed velocity
λ	-	shear modulus
μ	-	Lame constant
σ	-	Area density
e	-	Dielectric matrix
E	-	Electric field
v_p	-	Acoustic wave propagation velocity
ε	-	Piezoelectric matrix
p	-	Pitch
N	-	Number of pairs in the IDTs
l_{IDT}	-	The total length of the IDTs

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Due to the rapid growth of industrialization and the burning of fossil fuels and coal in the past decade, a substantial amount of hazardous gases have been released into the environment [1, 2]. Carbon dioxide (CO_2) is among these hazardous gases, which contributes to and is responsible for global warming, the greenhouse effect and rapid climate change. Additionally, it endangers human health by causing respiratory and nasal inflammation, fatigue, and headaches [2, 3]. To avoid such concerns, it is vital to regularly monitor CO_2 levels in the environment, and maintain a superior environmental health status and better air quality [4-6]. For the accurate quantification of the CO_2 gas, highly sensitive analytical techniques such as gas chromatography, spectrophotometry, and high-performance liquid chromatography have been considered. Despite their accuracy, these techniques have several salient drawbacks, such as high costs, lack of portability, and high-power consumption. Furthermore, the application of such techniques often requires complex and time-consuming pre-treatment steps and highly-skilled operators. These procedures cannot provide gas exposure information on a real-time basis. Consequently, microelectromechanical systems (MEMS) gas sensors have been introduced [7]. Compared to conventional gas sensing techniques, MEMS gas sensors have advantageous features such as higher sensitivity, lower power consumption, more compact designs, and significantly lower associated costs [8-10]. MEMS gas sensors can be classified under three primary types: electrochemical, optical, and acoustic. Each of type of sensor has a different operating mechanism for gas sensing. Among these sensors, MEMS surface acoustic waves (SAWs) have garnered considerable interest in the detection of gases, attributable to their improved sensitivity with high accuracy, fast response, and better stability [11]. The operating mechanism of earlier conventional SAW sensors comprise a pair of Interdigital transducers (IDTs) and one layer of metal-based sensing material in between the IDTs. These sensors have shown significantly lower performance and sensitivity, owing to the sensing elements that absorb gas molecules.

To improve overall sensitivity, SAW sensors with various nanostructured material-based sensing layers have been recently applied. Nanostructured materials show significant improvements in sensitivity, primarily due to their high surface-to-volume ratio, as well as enabling the design of a compact gas sensor. Therefore, the effort to incorporate the nanostructured material-based sensing layers into the SAW sensors may further improve their sensing capability.

1.2 Problem Statement

Numerous sensing technologies have been widely employed to accurately monitor and evaluate CO_2 levels [5, 12]. Despite their high precision, these techniques have several drawbacks, including low sensitivity and selectivity, abrasion of sensitive materials (especially for electrochemical sensors), high power consumption, difficulty in miniaturization, expensive costs, and lack of portability [5, 12, 13]. SAW gas sensors, on the other hand, have been widely applied because of their high sensitivity, portability, fast response time, and room temperature operation [14, 15]. The gas sensing mechanism of a SAW sensor primarily relies on the sensing element utilized to absorb gas molecules. Consequently, the sensing layer has a crucial role in SAW sensors. SAW sensors with various nanostructured materials-based sensing layers have recently been reported. Nanostructured materials show significant improvements in sensitivity, due to their high surface-to-volume ratio, as well as the optimal design of the compact gas sensor.

To date, polymers, metal oxides, and 2D material are among the sensing materials used in the sensitive layer of SAW sensors. Polymer-based sensitive materials have been widely reported as chemical warfare agents. However, polymer deposition on SAW sensors has a series of disadvantages, such as high losses and low propagation velocity due to moisture. In contrast, the inorganic metal oxide-based sensing material used in the SAW sensor has been reported to have high sensitivity only at high temperatures, and not at room temperature [19, 20]. Several researchers have proposed that improving sensitivity SAW sensors can be accomplished by using 2D material films as sensing materials in recent years. 2D materials has attracted considerable interest in the sensor industry, primarily due to its large surface area and capability for gas molecule adsorption. Although other 2D materials, such as CNT and graphene have been used in the gas sensor because of their higher sensitivity, higher surface-area-to-volume ratio and shorter response time. However, CNT material requires a purification process and lack of selectivity of raw CNT meanwhile higher quality graphene thin films still lack reproducibility and it is difficult to control the thickness material bi-layer or tri-layer Thus, it is necessary to develop a SAW gas sensor with high sensitivity and low-cost sensing material. Nonetheless, the long-term durability of the sensing material and its compatibility with complementary metal-oxide semiconductor devices for the large-scale production of SAW sensors remain elusive. Additionally, SAW gas sensors with highly sensitive sensing layers and low costs merit further empirical investigation.

1.3 Research objective

This work aims to design and fabricate a SAW gas sensor with a nanostructured Si-based sensing layer for detecting CO_2 gas. More specifically, this study aims to achieve the following research objectives:

1. To demonstrate the viability of Si nanostructures as a sensing material for SAW gas sensors of CO_2 gases by means of finite element analysis.
2. To design and fabricate a nanostructured Si sensing layer-based SAW gas sensor for CO_2 gas detection.
3. To experimentally characterize the performance of the developed SAW gas sensor in terms of sensitivity, time response and recovery.

1.4 Research scope

Scope of this work as follows:

1. The research only focuses on acoustic type gas sensors (SAW sensors) that comprise aluminium (Al) IDT, Lithium niobite ($LiNBO_3$) as a substrate and amorphous Si nanostructured as the sensing layer.
2. Design consists of 15 mm × 0.1 mm size of substrate, 200 um length of pitch and 3000 um length of sensitive material.
3. A metal-assisted chemical etching (MACE) process was used to fabricate the nanostructure on the substrate of SAW Sensors.
4. Due to its safety at lower concentrations, CO_2 was chosen as the test gas for the SAW sensor.

1.5 Research Significant

This work reports a novel gas sensor based on the SAW principle using nanostructured Si as the sensitive layer. The developed sensor provides improved sensitivity and time response, and operates at room temperature. Due to the morphology, highly specific surface area, and suitable gas diffusion material, it is expected that the nanostructured Si provides high sensitivity. This sensor also uses simple and cost-effective fabrication techniques to realize the sensing materials using the MACE technique. This work also covers all aspects of the development of the SAW sensor, including in an depth literature review, theoretical analysis, simulation, fabrication and characterization which may be used as a guide for new researchers.

1.6 Organization of thesis

This study focuses on the fabrication of a SAW sensor with a Si-nanostructured sensing material, and characterizes the properties of the SAW sensor, including the sensitivity, time response and recovery time. All the processes used in this study are described in detail. This work consists of five (5) chapters, starting the introduction of the research in Chapter 1. This chapter describes the introduction, problem statement objectives, scope, contributions and potential impact of this research. Chapter 2 provides an overview of the types of advanced MEMS gas sensing technologies. All the SAW fundamentals and all advanced MEM sensors, including working principles, fabrication, and their applications are described in this chapter. Chapter 3 describes the research methodology, including the workflow, all the parameters the model used for simulation, design and fabrication of the saw sensor and finite element analysis. Characterization and discussion of the Si nanostructure SAW sensor for CO_2 gas sensing are covered in Chapter 4. Lastly, in Chapter 5 provides a conclusion and some recommendations that merit further investigation in future work.

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APPENDIX A

LIST OF PUBLICATIONS

The thesis is based on the contributions that has been reported in following paper:

Index Journal:

1. **Asri, M. I. A**., Hasan, M. N., Fuaad, M. R. A., Yunos, Y. M., & Ali, M. S. M. (2021). MEMS gas sensors: a review. *IEEE Sensors Journal*. (**Q1, IF: 4.325**) (Part of Chapter 2)

2. **Asri, M. I. A**., Hasan, M. M. Nafea, Yunos, Y. M., & Ali, M. S. M. (2022). Nanostructured Silicon-based Surface Acoustic Wave Sensor for CO_2 Gas Detection. *IEEE Sensors Journal*. (**Q1, IF: 4.325**) (Accepted) (Part of Chapter 3,4, and 5)

3. **Asri, M. I. A**., Hasan, M. N., Fua'ad, M. R. A., Abd Hamid, F. K., Yunos, Y. M., & Ali, M. S. M. (2021). Finite Element Analysis of Silicon Nanowire Array based SAW Gas Sensor. *ELEKTRIKA-Journal of Electrical Engineering*, *20*(2-2), 36-40. **(MyCite)** (Part of Chapter 3)

Indexed Conference Proceedings

1. **Ahmad Asri, M. I**., Hasan, M. N., Md Yunos, Y., & Mohamed Ali, M. S. (2022). Design and Analysis of Silicon Nanowire Array Based on SAW Gas Sensor. In *Control, Instrumentation and Mechatronics: Theory and Practice* (pp. 617-626). Springer, Singapore. **(SCOPUS Indexed)** (Part of Chapter 3)

2. **Asri, M. I. A., Hasan**, M. M. Nafea, Yunos, Y. M., & Ali, M. S. M. (2022). Silicon Nanostructure based Surface Acoustic Wave Gas Sensor. IEEE Sensors Conference Dallas USA, 2022. **(SCOPUS Indexed)** (Accepted) (Part of Chapter 3,4, and 5)