INVESTIGATION ON THICKNESS OF THIN FILM GAS SENSOR FOR HIGH SENSITIVITY GAS SENSOR

YAP PEI YEUAN

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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YAP PEI YEUAN

This report is submitted in partial fulfilment of the requirements for the degree of Bachelor of Electronic Engineering with Honours

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DECLARATION

I declare that this report entitled “Investigation on thickness of thin film gas sensor for high sensitivity gas sensor ” is the result of my own work except for quotes as cited in the references.

Signature : .....

Author : Yap Pei Yeuan .....

Date : 21 June 2022 .....

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Bachelor of Electronic Engineering with Honours.

Signature : .....

Supervisor Name : Dr. Siti Amaniah binti Mohd Chachuli

Date : 20 June 2022

DEDICATION

I specially dedicated to my supervisors, lecturers, family and friends, who always guide and support me to complete my final year project successfully.

ABSTRACT

VOC gas is a colourless and flammable gas. Hence, it is one of the main sources that causes combustion in high-temperature conditions. Besides, it is harmful to the health of living things. It will induce dizziness, headaches, vomiting, nose, eye, and throat irritation in the short term. In the long term, it will cause central nervous system damage, cancer, liver and kidney damage. As a result, a thin film gas sensor is used to detect the presence of VOC gases in the environment, although in a small region. A thin film gas sensor works on the premise of converting chemical quantities contained in gas into an electrical signal, such as current. The purpose of this project is to investigate the high sensitivity of thin film gas sensors at various thicknesses of the fabricated thin film. The substrates used are glass and Kapton film. There are several types of solutions used which consist of three different types of solvent (acetone, ethanol, and DI water) and three different concentrations of graphene power (0.01 g, 0.02 g, and 0.05 g of graphene power). The thickness of the thin film can be variable through different amounts of graphene power used in the solutions. All the prepared solutions are sonicated for 30 minutes to ensure they are well mixed. First, the electrode is screen-printed onto the substrates before being annealed at 150 degrees Celsius for 10 minutes. After that, a thin film is deposited on the substrate that contain

electrode by applying the dropping process with one drop of solution and undergoing annealing at 150 degrees Celsius for 10 minutes. The resistance-containing thin film gas sensor will next be used to test the target VOC gases. The VOC gases used are acetone and ethanol. Finally, the results of the gas test are used to perform further research. The gas sensors that are fabricated on the Kapton film substrates with 0.01 g graphene and 10 g DI water have high sensitivity to acetone and ethanol, where sample D1a has a sensitivity of 3.24% and D1b has a sensitivity of 7.02%. Sample E5b has the shortest response time (1 s), whereas sample D-5b has the quickest response time (5 s) of all the samples. The majority of fabricated gas sensors are more sensitive to acetone than to ethanol gas.

ABSTRAK

Gas VOC adalah gas tanpa warna dan mudah terbakar. Oleh itu, ia adalah salah satu sumber utama yang menyebabkan pembakaran dalam keadaan suhu tinggi. Selain itu, ia berbahaya kepada kesihatan makluk hidup. Ia akan menyebabkan pening, sakit kepala, muntah, hidung, mata dan kerengsaan tekak dalam jangka masa pendek. Dalam jangka masa Panjang, ia akan menyebabkan kanser, kerosotan system saraf pusat dan buah pinggang. Oleh itu, sensor gas filem nipis digunakan untuk mengesan kehadiran gas VOC di alam sekitar walaupun di Kawasan kecil. Sensor gas filem nipis berfungsi di premis menukar kuantiti kimia yang terkandung dalam gas menjadi isyarat elektrik, seperti arus. Tujuan projek ini adalah untuk menyiasat sensitivity tinggi sensor gas filem nipis pada pelbagai ketebalan filem nipis yang dibuat. Substrat yang digunakan adalah kaca dan filem Kapton. Terdapat beberapa jenis larutan yang digunakan, ia terdiri daripada tiga jenis pelarut yang berbeza (aseton, etanol, dan air DI) dan tiga kepekatan graphene yang berbeza (0.01g, 0.02g dan 0.05g graphene). Ketebalan filem nipis boleh berubah-ubah melalui jumlah kuasa graphene yang berbeza yang digunakan dalam larutan. Semua larutan yang disediakan akan menjalani sonikasi selama 30 minit untuk memastikan ia bercumpur dengan baik. Dalam proses pertama, elektrod dicetak skrin ke substrat sebelum dilil pada 150 darjah Celsius

selama 10 minit. Selepas itu, filem nipis disimpan pada substrat yang mengandungi elektrod dengan proses “dropping” dengan satu titisan larutan dan menjalani penyepulindapan pada 150 darjah Celsius selama 10 minit. Sensor gas filem nipis yang mengandungi rintangan akan diguji dengan gas VOC. Gas VOC yang digunakan adalah aseton dan etanol. Akhirnya, keputusan ujian gas digunakan untuk melakukan penyelidikan selanjutnya. Sensor gas yang dibuat pada substrat filem Kapton dengan 0.01 g graphene dan 10 g air DI mempunyai sensitivity yang tinggi terhadap aseton dan etanol, di mana sampel D1a mempunyai sensitivity 3.24% dan D1b mempunyai sensitivity 7.02%. Sampel E5b mempunyai masa tindak balas terpendek ketika gas disalurkan (1 s), manakala sampel D-5b mempunyai masa tindak balas terpantas ketika pelepasan gas (5 s) berbandingkan semua sampel. Sebilangan besar sensor gas yag dibuat lebih sensitive terhadap aseton daripada etanol.

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I am grateful to have the opportunity to conduct this project. First of all, I would like to thank University Technical Malaysia Malacca (UTeM) for giving me the chance to conduct the final year project. Besides, I would like to express my gratitude to my main supervisor (Dr. Siti Amaniah binti Mohd Chachuli) and co-supervisor (Dr. Muhammad Idzdihar bin Idris) for giving me insightful suggestions, advice, and inspiration throughout my project. Moreover, I would like to express my gratitude to my family and friends who guided and supported me to complete my project.

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LIST OF SYMBOLS AND ABBREVIATIONS

DI water	:	Deionized water
Gr	:	Graphene
IDEs	:	Interdigitated electrodes
IPA	:	Isopropyl alcohol
SEM	:	Scanning electron microscope

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CHAPTER 1

INTRODUCTION

1.1 Project Background

Volatile organic compounds (VOCs) are emitted as colourless gases from certain liquids or solids. VOC gas covers a wide range of chemical substances that are naturally occurring or man-made. There are common types of VOC gas, including acetone, acetic acid, acetylene, benzene, ethanol, formic acid, methanol, isopropanol, and toluene. VOC gases come from building materials (such as carpet, paint, and composite wood products), personal care products (like cosmetics, nail removers, and hand sanitizers), and daily used equipment (like cooking gas, fuel oil, and dry cleaning). VOC gas has a high vapour pressure and is flammable. So, it is one of the main causes of firebreaks. Moreover, VOC gases will have a negative effect on living things' health. If we are exposed to VOC gas in the short term like hours to days, we might experience headaches, vomiting, dizziness, worsening of asthma symptoms,

eye, nose, and throat irritation. But chronically exposed to it might cause cancer, central nervous system damage, liver and kidney damage. A gas sensor is an electronic device to identify and detect different types of gases. A thin film gas sensor has a very fine layer of around 10 nm to 1µm deposited on a substrate. Therefore, this project will fabricate thin film gas sensors with varying thicknesses, test them with several types of VOC gases, and investigate the results.

1.2 Problem Statements

Volatile organic compounds (VOCs) are flammable gases, so it is easy to cause combustion when exposed to high temperatures. According to ACS' Environmental Science and Technology researchers, they have analysed the level of particulate matter and VOCs surrounding firefighters actively fighting fires, finding the highest exposures among hotshot teams and those establishing firebreaks [1]. Moreover, VOC gas will affect living things' health. According to the Minnesota Department of Health, exposure to high levels of VOC gas in acute terms (hours to days) may cause us headaches, dizziness, worsening of asthma symptoms, vomiting, eye, nose and throat irritation [2]. While we exhibit VOC gas at a high percentage chronically (years to a lifetime), we might have some symptoms like cancer, central nervous system damage, liver and kidney damage [2]. Moreover, some VOC gases cause cancer in animals, according to the United States Environmental Protection Agency [3].

1.3 Objectives

i. To fabricate the thin film gas sensor on different substrates using the dropping technique.

ii. To investigate the various thicknesses of thin film gas sensors for the target VOC gases.

iii. To analyse the performance of the fabricated thin film gas sensors in terms of sensitivity, response time, and recovery time.

1.4 Project Scopes

The substrates used in this project are glass and Kapton film. The substrate is coated with various types of solutions with three different types of solvent (acetone, ethanol, and DI water) and several amounts of graphene. The different amounts of graphene power (0.01g, 0.02, and 0.05g) are used to obtain various thicknesses of thin film. All the measurements of the samples are done on an analytical balance. Then, the solutions are sonicated using an ultrasonic cleaner for 30 minutes. First, the screen-printing technique is used to fabricate an electrode on the substrate with silver paste. Besides, the dropping method is used to form thin film layers on the substrates. A scanning electron microscope (SEM) is used to observe the structure of crystalline graphene in the thin films on the substrates, which are glass and Kapton film. Silver paste is pasted with 2.5cm of copper wire. It acts as a connecter to make the thin film have a connection to become a gas sensor. A picoammeter is a tool used to check the current contained in the gas sensor by applying a certain voltage to it. The well-performed thin film gas sensors are tested with the targets of VOC gases, which are acetone and ethanol. Finally, analyse the sensitivity, response time, and recovery time of the thin film gas sensor through the graph and some calculations.

CHAPTER 2

BACKGROUND STUDY

2.1 Introduction

This chapter is to review and cite literature documents such as journals from researchers about the thin film gas sensor to detect VOC gases. Besides, a VOC detector is an essential piece of personal protective equipment (PPE) for detecting and monitoring exposure to these types of dangerous VOC gases. The thin film gas sensor has a limitation in thickness where the film is below 1μm. This session includes the theory of thin film gas sensors, VOC gas sensors in the market and from researchers, sensing materials that can be used to produce VOC thin film gas sensors, thin film deposition techniques used by researchers, and characteristics of thin film gas sensors.

2.2 Chemical-based Thin Film Gas Sensor

A chemical sensor is an analyser that responds to a single analyte in a reversible and selective manner and converts input chemical quantities, ranging from the concentration of a specific sample component to a whole composition analysis into an analytically electrical signal [4] as illustrated in Figure 2.1. This type of chemical sensor is mostly used in emerging discipline such as biology, chemistry, electricity, optics, acoustics, thermology, semiconductor technology, microelectronic technology, and so on [4].

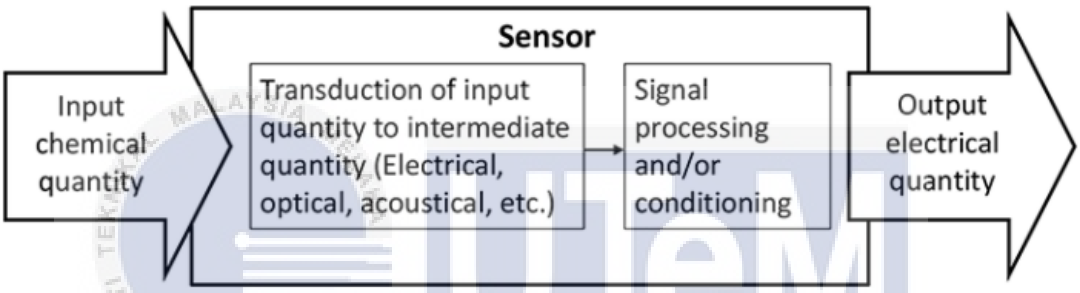


Figure 2.1: The schematic diagram of sensor principle [4].

A thin film has a thickness of film that is less than 1 μm [5]. It is formed when a solid substance is deposited on a solid support, known as a substrate by using either a physical or chemical method. It can be produced by using nanostructure technology. By using nanostructured sensing materials, nanotechnology can help to develop the next generation of gas sensor devices with improved sensing performance in terms of fast response and recovery time, low power consumption, good reversibility, and excellent sensitivity at extremely low gas concentrations [5]. Examples of nanostructure gas sensors are semiconducting metal oxide nanostructure gas sensor, polymer-based nano sensor, carbon-based nano sensor, and surface acoustic wave-based thin film gas sensor.

2.2.1 Semiconducting Metal Oxide Nanostructure Gas Sensor

Semiconducting metal oxides are the most commonly used material in fabricating nanostructured gas sensors. This type of gas sensor will change its electrical conductivity when its surface is exposed to a detecting gas. The schematic diagram of the metal oxide thin film gas sensor is illustrated in Figure 2.2, which consists of nanowire, nanobelt, and nanoparticles. Nanostructure metal oxide thin film has high sensitivity surface activity, high adsorption of gas species on the surface and subsequent catalytic activity with the absorbed species improves sensing performance and thereby reduces response time. Examples of semiconducting metal oxide thin film gas sensor are zinc oxide thin film gas sensor, and tin (iv) oxide thin film gas sensor.

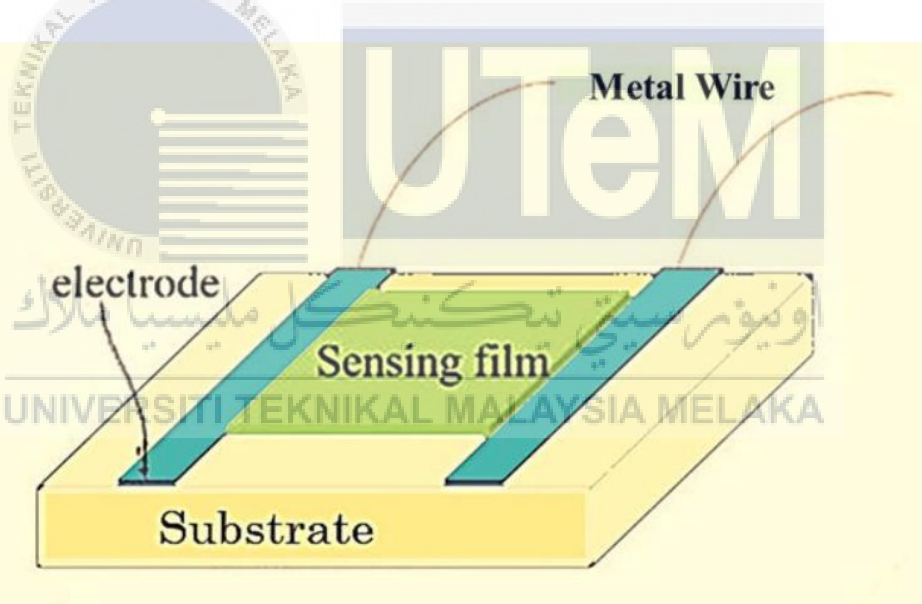


Figure 2.2: Schematic diagram of metal oxide thin film gas sensor [5].

2.2.2 Polymer-based Nanosensor

An organic polymer-based nanosensor is commonly used for detecting medicine, aromatic compounds, organic vapour, gases, ionic species, and amines [5]. Normally, conductive polymers are deposited as a thin layer on a substrate [5]. To monitor sensing current, the sensor structures can be either a field-effect transistor with a regulated electric field or a film with two electrodes [5]. The detecting area of the gas sensor and the thickness of the film will influence the sensitivity of the polymer-based gas sensor [5]. To increase its sensitivity, carbon nanoparticles, metal oxides, and fibres are added to the polymer-based sensors [5]. This deposition technique is cheap but polymer-based sensor lacks selectivity and stability with time and temperature [5]. Although adding the fibers and nanoparticles to the sensor but the stability and selectivity but still incompletely been solved [5].

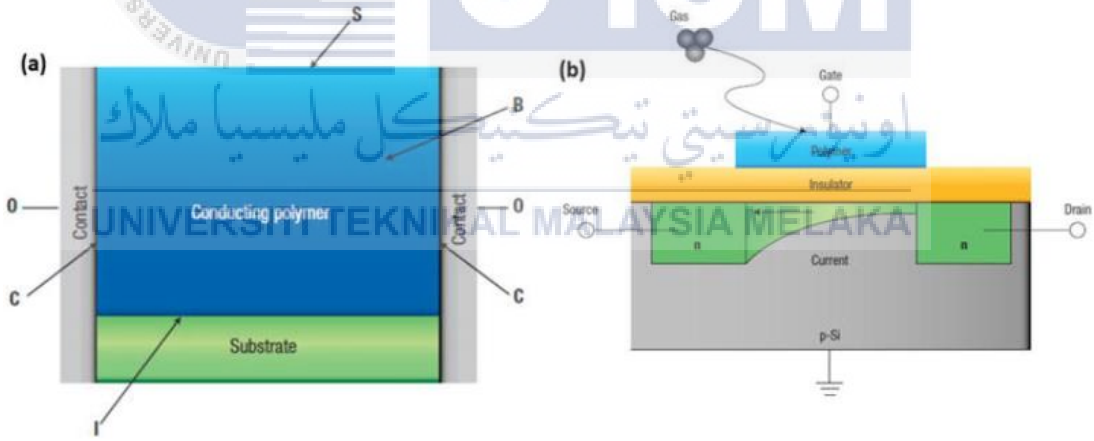


Figure 2.3: (a) shows the chemiresistor device structure where S = surface, I = interface with insulating substrate, C = contact. (b) shows the conducting polymer active layer in field-effect transistor (FET) [5].

2.2.3 Carbon-based Nanosensor

Carbon-based materials have been shown to be particularly promising gas sensors due to their high surface area, good thermal and chemical durability, and outstanding intrinsic electrical characteristics compared to other nanostructured gas sensors [5]. Figure 2.4 shows the schematic diagram of the carbon-based sensor where it consists of a source, a drain, and a CNT on the substrate.

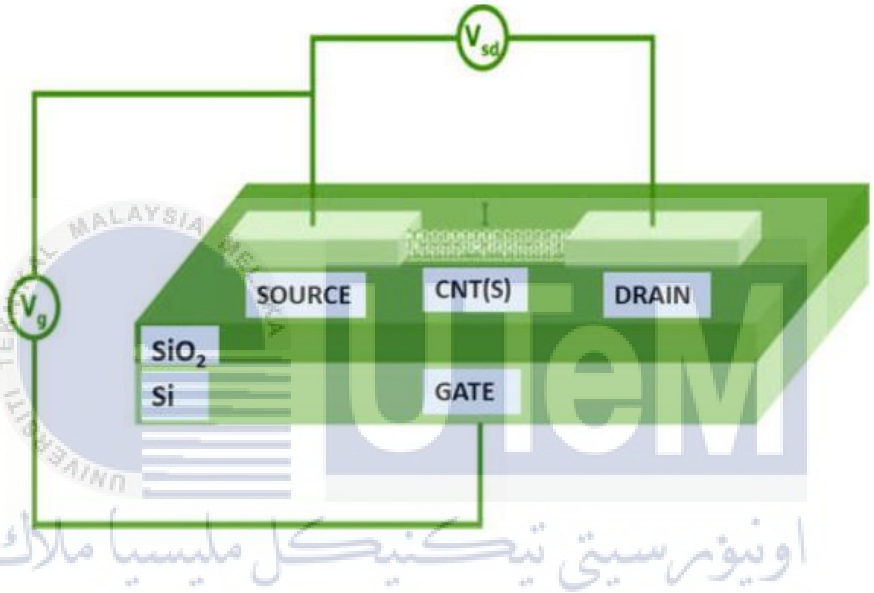


Figure 2.4: The schematic diagram of a FET device based on CNT [5].

2.2.4 Surface Acoustic Wave-based Gas Sensor

A surface acoustic wave (SAW)-based gas sensor has fast response, high sensitivity, and a small volume compared to electrochemical sensors [6]. Figure 2.4 shows a conventional SAW gas sensor with a delay line-patterned SAW device and a selective gas-sensitive thin-film coated along the SAW propagation channel between the two interdigital transducers (IDTs) [6]. Depending on the physical class of the sensitive thin-film, adsorption influences SAW propagation via a viscoelastic, so-called mass loading or acoustic-electric effect [6].

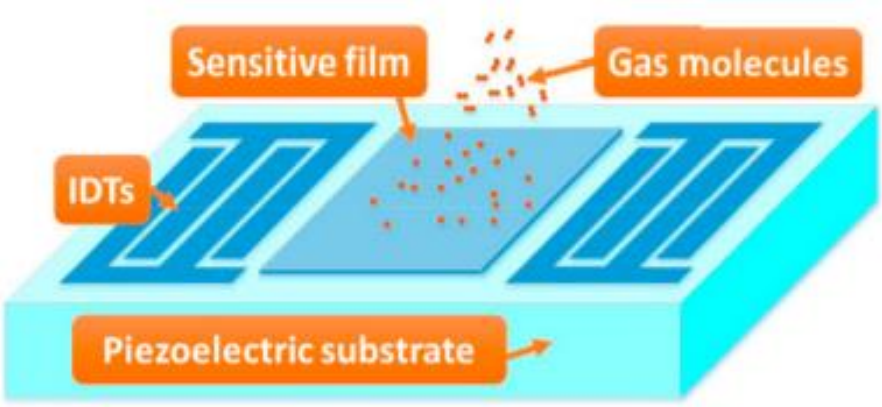


Figure 2.5: The schematic diagram of SAW gas sensor [6].

2.3 VOC Gas Sensor in Market

A MiniPID 2 PPM VOC gas sensor is one of the high sensitivity gas sensors to detect VOC gases that are sold in the market. The range of VOC gas that can be sensed is more than 4000 ppm [7]. Its sensitivity can be achieved at greater than 0.65 mV/ppm [7]. Its minimum detection limit can be achieved at up to 100 ppb [7]. Its response time to VOC gas is less than 3 s [7].

Besides, a MiniPID 2 HS is used to sense the amount of VOC gases in the surrounding environment. It can detect VOC gases in the range of 0 ppm up to 3 ppm [8]. The sensitivity is more than 600 mV/ppm [8]. Its response time will be longer than MiniPID 2 PPM, which is less than 12 s [8]. The minimum detection limit is 0.5 ppb [8].

Moreover, a Falco pumped fixed VOC gas monitor is used to detect a wide range of VOC gases in the amounts of ppm and ppb. It can directly display the amount of surrounding VOC gases in ppm with 3 decimal places. It is the biggest in terms of size and heaviest compared to the MiniPID 2 PPM sensor and MiniPID 2 HS, which have a combined weight of 3.3 kg [9]. It has four detection ranges, which are 0 – 10 ppm, 0 – 50 ppm, 0 – 1000 ppm, and 0 – 3000 ppm [9].

Table 2.1: The characteristic of each gas sensor sells in market.

Name of Gas Sensor	Sensitivity	Detection Range	Minimum Detection Limit	Maximum Response Time (T_{90})	Power Consumption	Weight and Dimension
MiniPID 2 PPM	0.65 mV/ppm	within 4000 ppm	100 ppb	3 s	100 mW	16.5 mm (h) × 20 mm (d)
MiniPID 2 HS	600 mV/ppm	0 – 3 ppm	0.5 ppb	12 s	100 mW	16.5 mm (h)×20 mm (d)
Falco pumped fixed VOC gas monitor	50.0 ppm, 0.01 ppm	0 – 100 ppm, 0 – 50 ppm, 0 – 1000 ppm, and 0 – 3000 ppm	0 ppm	10 s	Typical 2 W, maximum 7 W	3.3kg, 291 mm (h) × 125 mm (d)

2.4 Development of VOC Thin Film Gas Sensor

The thin film gas sensors can be investigated from the experiments that have been conducted by researchers via the journal. Table 2.2 shows the material and deposition technique used to form a thin film gas sensor, which was done by researchers to detect the existing VOC gases.

Table 2.2: The feature of thin film gas sensor from journal.

Material	Deposition Technique	Substrate	Thickness of Thin Film Gas Sensor	Type of Gas Tested	Sensitivity	Response Time	Recovery Time	Reference
$Cu_{1-x}Zn_xO$	Spray pyrolysis	Soda lime glass substrate	600nm	Acetone	-	100 ppm: 40s 200 ppm: 37s 300 ppm: 26s 400 ppm: 22s 500 ppm: 20s	100 ppm: 33s 200 ppm: 49s 300 ppm: 83s 400 ppm: 132s 500 ppm: 154s	[10]
				Ethanol	1 ppm: 4% 5 ppm: 11% 10 ppm: 18%	100 ppm: 24s 200 ppm: 28s 300 ppm: 30s	100 ppm: 300s 200 ppm: 238s 300 ppm: 209s 400 ppm: 170s 500 ppm: 117s	

						400 ppm: 34s 500 ppm: 40s		
Gallium nitride	Spin coating	Silicon	-	Nitrogen	150 sccm: 50%	-	-	[11]
				Hydrogen	150 sccm: 60%	-	-	
Indium (III) sulfide	Spray pyrolysis	SiO_2 / Si	-	Ethanol, methanol, isopropanol, acetone, and toluene	-	-	-	[12]
Methylammonium lead iodide	Spin-coating	Glass	100nm, 250nm, 300nm	Acetone, acetonitrile, benzene, chloroform, ethanol, hexane, isopropanol, methanol, toluene	-	-	-	[13]
Perovskite oxides	Thermal evaporation	STO	-	Ethanol	-	50 ppm: 54s	50 ppm: 23s	[14]
Reduced graphene oxide	Drop casting	SiO_2 / Si	200nm	Acetone	-	100 ppm: 23s	100 ppm: 20s 500 ppm: 30s	[15]

						500 ppm: 25s		
RGO, CuNW-RGO	Dropping	-	-	Benzene, hexane, acetone	100 ppm: 0.5% 500 ppm: 1.8%	-	-	[16]
Tin(IV) oxide	Spin-coating	Thermally oxidized silicon wafer	10nm – 25nm	Ethanol	-	1000 ppm with 84℃: 13s	1000 ppm with 84℃: 4s	[17]
Zinc oxide	E-beam evaporation	SiO_2 / Si	100nm	Formaldehyde, acetic acid, acetylene, formic acid, toluene, benzene, acetone, ethanol, methanol, isopropanol	-	-	-	[18]
Zinc oxide	RF reactive sputtering	Silicon wafer	250nm	Acetone	100 ppm: 30%	-	-	[19]

2.5 Sensing Material in VOC Thin Film Gas Sensor

The sensing materials used in fabricating a thin film gas sensor by the research are listed below. The structure of the material will be discussed in this session.

2.5.1 Graphene

Graphene is a carbon-based substance made up of carbon atoms linked together in a hexagonal arrangement with two-dimensional [20]. Graphene has a strong electrical conductivity of 10^6 S cm^{-1}, strong thermal conductivity of 5000 W $m^{-1}k^{-1}$. Besides, graphene has a large surface area of 2630 m^2g [21].

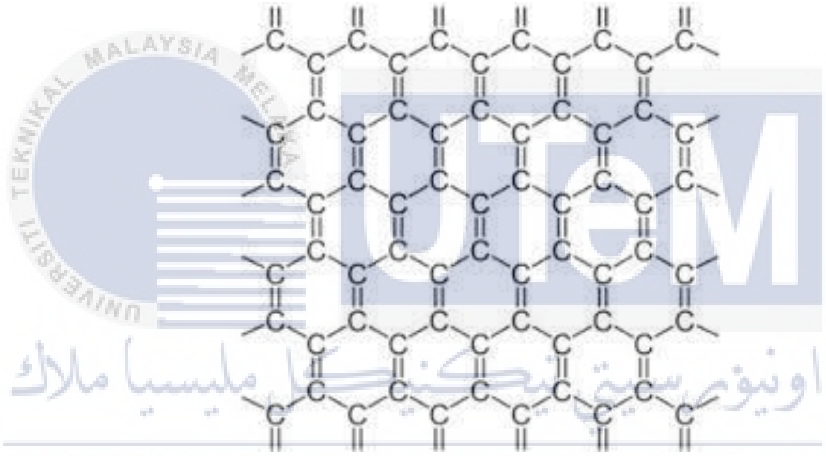


Figure 2.6: The structure of graphene [22].

2.5.2 Reduced Graphene Oxide

Reduced graphene oxide (rGO) is a type of graphene oxide that has been processed by several methods through chemical, thermal, and other methods to reduce the oxygen content [23]. The surface area of rGO is 110.1616 m^2g [24]. The electrical conductivity of rGO is 6300 S cm^{-1} [25].

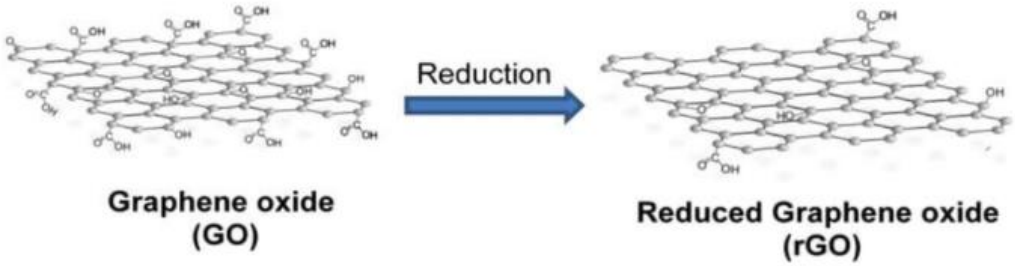


Figure 2.7: The structure reduced graphene oxide from graphene oxide [25].

2.5.3 Tin (IV) Oxide

Tin (IV) oxide is an inorganic compound with an off-white or white crystalline power or solid. It is insoluble in water but soluble in concentrated hydrochloric acid and sulfuric acid [26]. It has a density of 6.95 g cm^{-3} [26].

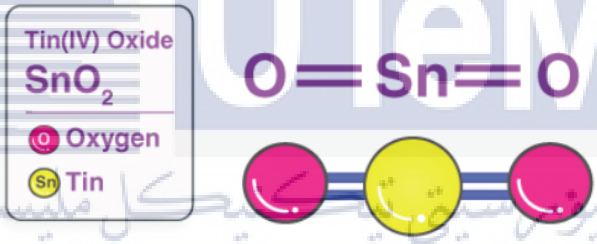


Figure 2.8: The structure tin (IV) oxide [27].

2.5.4 Zinc Oxide

Zinc oxide is a white, powdery mineral inorganic compound. It is commonly used in cosmetics and personal care products such as lotions, makeup, nail products, and sunscreen products [28]. The zinc oxide nanoparticles are produced with a surface area of 88.89m^2g^{-1}, and a pore volume of 0.98 cm^3g^{-1} [29].

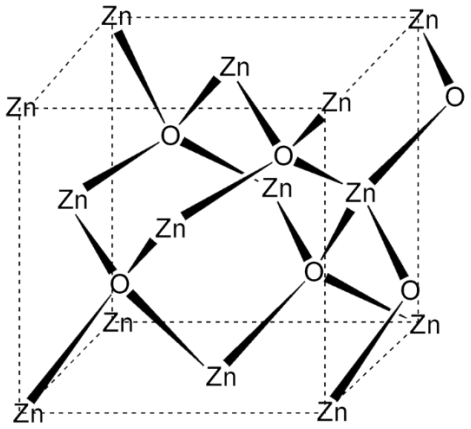


Figure 2.9: The structure zinc oxide [30].

2.6 Thin Film Deposition

Deposition on the thin film gas sensor is an essential process to use to form a thin film. There are two categories of thin film deposition methods, which are physical deposition and chemical deposition. Some techniques will be discussed in this session. Evaporation and sputtering techniques are examples of using physical deposition. Examples of chemical deposition are sol-gel (such as alkoxide precursors in organic solvents, dip coating technique, and spin-coating technique) and chemical bath deposition technique.

2.6.1 Drop Casting Technique

Drop casting is the simple technique used to produce thin films without specific equipment [31]. The thickness of a thin film and its properties depend on the concentration and volume of the dispersion [31]. The steps for drop casting are dropping, evaporation, and drying as shown in Figure 2.10. Less material wastage is the benefit of using this method. The disadvantage of this method is that it is difficult to form a uniform thin film layer and to control its thickness [31].

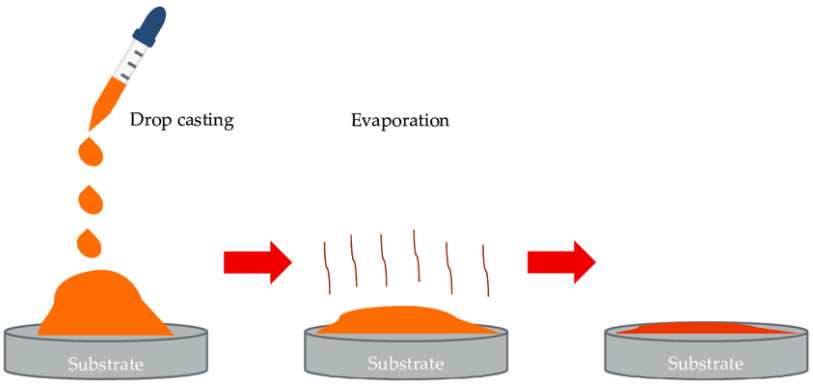


Figure 2.10: The drop casting process [32].

2.6.2 Spin Coating Technique

Spin coating is a commonly used technique to deposit a uniform thin film using a solution on a small substrate in a fast way. Spin-coating has four main steps in this process, which are deposition, spin up, spin off, and evaporation. Figure 2.11 shows the illustrations for the four major procedures of spin coating. Deposition is the first step in spin-coating in which the solution is dispensed onto the substrate surface. Next, it will undergo a spin up process. Once the solution is translated to the substrate surface, the rotation disc in the spinner speeds up either gradually or immediately speeds up according to the desired rotation speed [33]. Spin off is the third step in this technique. During the third stage, the solution begins to turn into a thin film as the solution is flung off the substrate [33]. Lastly, is the evaporation process. In this process, the solvent evaporates to form a finer thin film, and its evaporation rate is dependent on volatility, solvent vapour pressure, and ambient temperature [33].

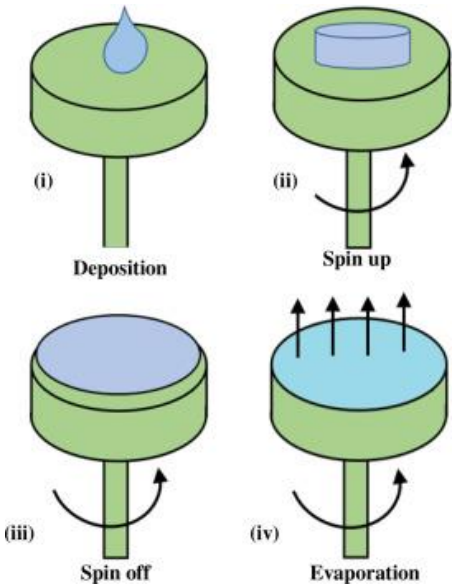


Figure 2.11: The four main steps of spin coating [34].

2.6.3 Sputtering Technique

Sputter deposition is a widely used technique to deposit thin films on the substrate [35], also known as a high vacuum-based coating technique that belongs to the group of physical vapour deposition (PVD) processes [36]. Sputtering works by pulling the atoms of a substance one by one and depositing them on the substrate using the energy of a plasma, which is a partially ionised gas on the cathode (the surface of the target) [36]. A plasma is formed by ionising a pure gas (typically Argon) using a potential difference (pulsed DC) or electromagnetic excitation (MF, RF). The plasma is made up of Ar^+ ions that are accelerated and confined around the target owing to the existence of a magnetic field [36]. By impacting the target, each ionised atom transmits its energy and rips an atom with enough energy to be transferred to the substrate [36]. Although the plasma is formed at high pressures, which range from 10-1 to 10-3 mbar, it is required to start at a lower pressure before adding Argon to minimise contamination from remaining gases [36]. There are several types of

sputtering that can be used to deposit the thin film, which are DC diode sputtering, RF sputtering, DC triode sputtering, magnetron, and reactive sputtering.

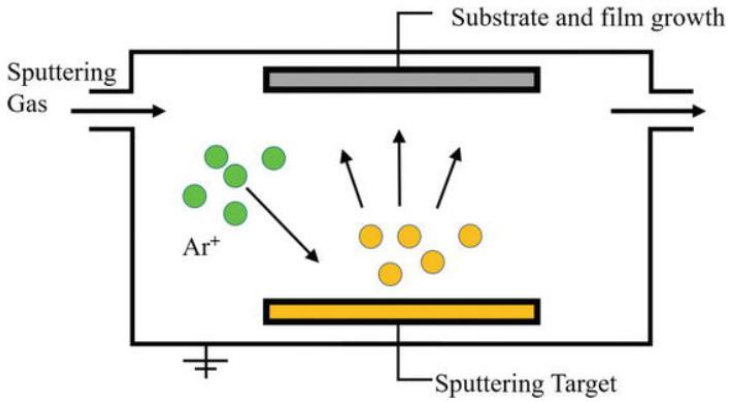


Figure 2.12: The four main steps of spin coating [34].

2.7 Characteristic of Thin Film Gas Sensor

The performance of thin film gas sensors can be evaluated by several parameters, which are analysing the sensitivity, response time, and recovery time for thin film gas sensors are the important elements to determine which thickness of the thin film gas sensor has the highest sensitivity to achieve the aim of this project.

2.7.1 Sensitivity

The sensitivity of the thin film gas sensors can be calculated by using the below formula:

$$S = \frac{|R_a - R_g|}{R_g} \times 100\% \quad \text{..... (2.1)}$$

where S represents sensitivity of a thin film gas sensor in percent, R_a represents resistance of a gas sensor in air, and R_g represents resistance of gas sensor in gas atmosphere [10].

2.7.2 Response Time

Response time is the time difference between the change of the component to measure a system response from 90% of the final reading to 10% of the original reading [37]. Figure 2.13 shows an example of obtaining response time (t_{90}) from the graph.

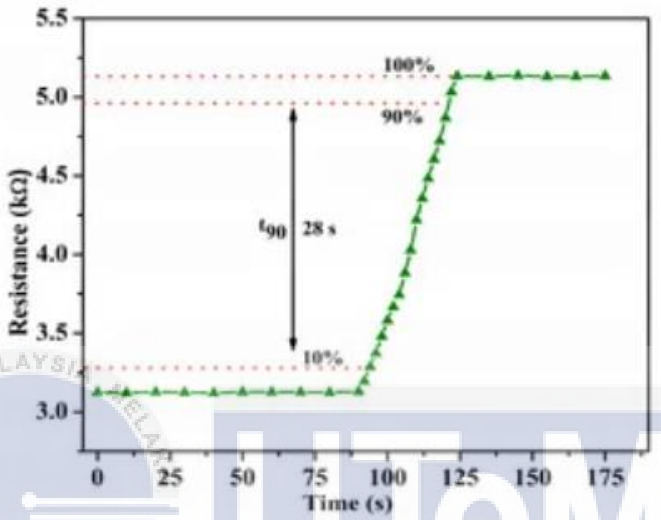


Figure 2.13: The example of response time ($\mathbf{t_{90}}$) from the graph which is 28 s [37].

2.7.3 Recovery Time

The recovery time is defined as the time taken for a sensor to return from 90% of the exposed gas time to 10% of the original baseline signal after removing the target gas [37]. Figure 2.14 shows an example of recovery time based on the graph.

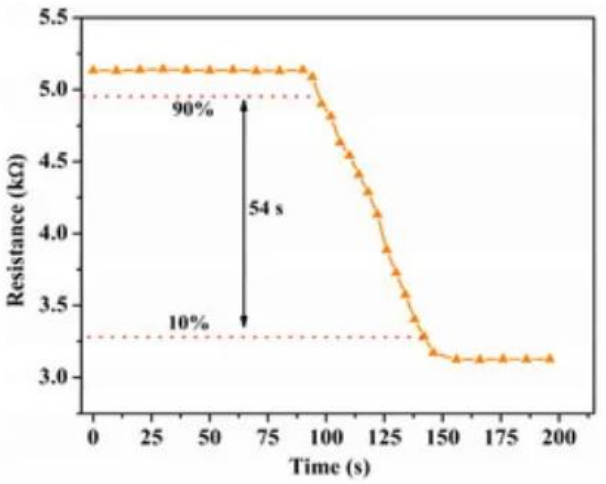


Figure 2.14: The example of recovery time from the graph is 54 s [37].

CHAPTER 3

METHODOLOGY

3.1 Introduction

The flow of a project can be divided into three sections. The first section is to fabricate thin film gas sensors in various thicknesses. The second section is to test the well performance thin film gas sensor with a variety of VOC gases. The third section is to analyse the sensitivity, recovery time, and response time of thin film gas sensors of various thicknesses. Figure 3.1 depicts the flow chart for a suitable method of thin film gas sensor deposition in the project.

3.2 Overall Project Flow Chart

First, the samples are prepared using the purchased materials. After completing the preparation, screen-print the electrode onto the substrate and anneal it in an oven to ensure it is fully dry. After that, a dropping method is used to form a thin film on the electrode. Apply a second annealing treatment to the thin film that has been deposited to make it more stable. A picoammeter is used to measure the current of a thin film gas sensor by applying a certain voltage to obtain the IV graph once the fabrication process is completed. This process can be used to identify whether it functions properly or otherwise. If the IV graph shows a straight horizontal line, it means it cannot be used, and the fabrication process needs to be reconstructed. They can proceed to expose themselves to VOC vapours if their resistance is detectable by using a tool called a picoammeter. The crystallinity of the thin film is assessed using scanning electron microscopy (SEM). If the thin film does not include crystallinity particles during the checking, it cannot be employed, and it requires starting over the same procedure starting from sample preparation. If crystallinity particles are discovered during the characterisation process, the thin film can be used to test with VOC gases. The thin film gas sensor will then be exposed to various VOC gases and readings will be recorded. Finally, we analyse the response time, recovery time, and sensitivity at various thicknesses of gas sensors.

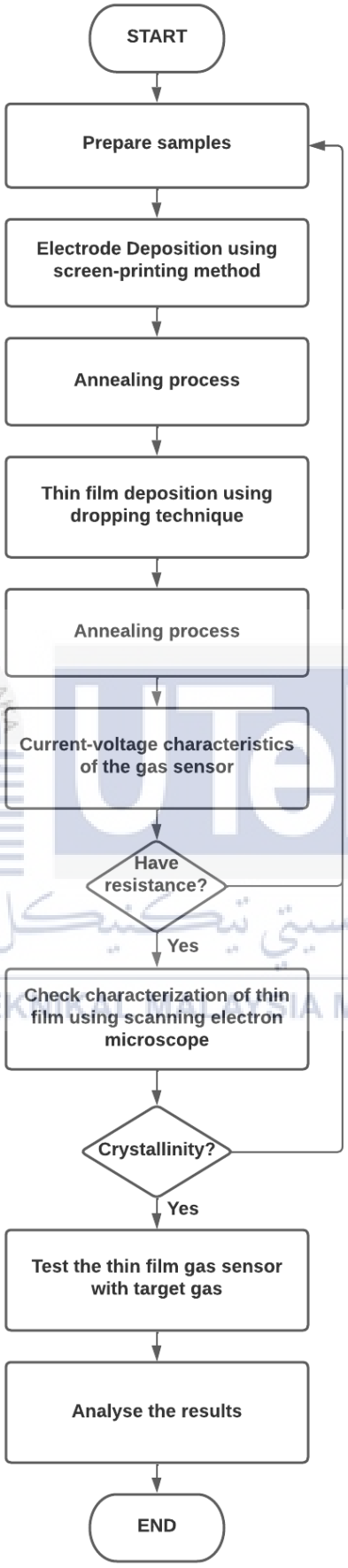


Figure 3.1: Flow chart of the project.

3.3 Fabrication Process of Thin Film Gas Sensor

Fabrication is an essential step in producing a thin film gas sensor that can detect VOC gas in the environment. The electrode is initially applied to the substrates by a screen-printing process. After that, it undergoes annealing for 10 minutes using a 150 ℃ oven. Then, apply the dropping technique to form a thin film deposit on the electrode. The various thicknesses can be formed by adjusting the amount of graphene power used in the solution. The more graphene power is used in the solution, the thicker the thin film will form on the substrate. Next, it goes through annealing for the second time at the same temperature and duration of time. Then, add about 2.5 cm of copper wires at the two ends of the electrodes, and anneal for third time at the same time and temperature to obtain a thin film gas sensor. The fabrication is illustrated in Figure 3.2.

Figure 3.2: Flow diagram of fabrication process of thin film gas sensor.

3.3.1 Preparing of Substrates

There are two types of substrates used in this project, which are glass and Kapton film. The cutting and cleaning processes will be done on the substrates before moving on to the fabrication process.

3.3.1.1 Glass

The glass slides are cut into 1.5 cm × 2.0 cm. The completed cut glasses are shown in Figure 3.3. The glasses are next fully immersed in isopropanol (IPA) solvent as illustrated in Figure 3.4. After that, an ultrasonic cleaner is used to sonicate them for 20 minutes. The IPA solvent in the beaker is then poured into a waste container and dried for 30 minutes in an oven at 80 ℃.

Figure 3.3: The glasses with 1.5 cm × 2.0 cm.

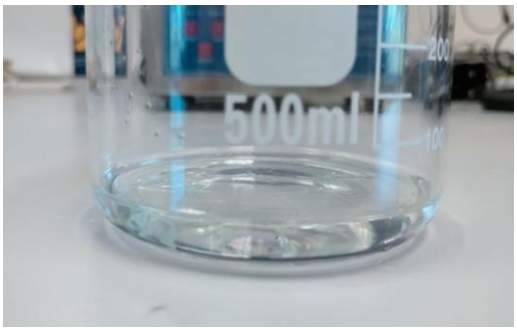


Figure 3.4: The glasses are fully soaked in IPA solvent.

3.3.1.2 Kapton Film

First, the Kapton films are cut into 1.5 cm × 2.0 cm as illustrated in Figure 3.5. To ensure that all Kapton films are totally clean, they are wiped using isopropanol solvent.

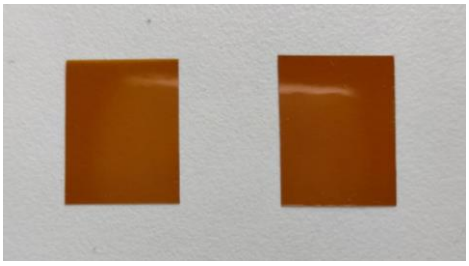


Figure 3.5: The Kapton films with 1.5 cm × 2.0 cm.

3.3.2 Electrode Deposition

The electrodes are deposited on the surface of substrate using a screen-printing process. To create an electrode with the area of 1 cm × 1 cm, a screen-printing board is placed on the substrate, and then silver paste is applied to the board and pressed onto the substrate using a squeegee. In both electrode production methods, copper wires with a length of around 2.5 cm are employed. Then, it will be annealed in a 150 ℃ oven for 10 minutes. The entire fabricated electrode using the screen-printing method is illustrated in Figure 3.6.

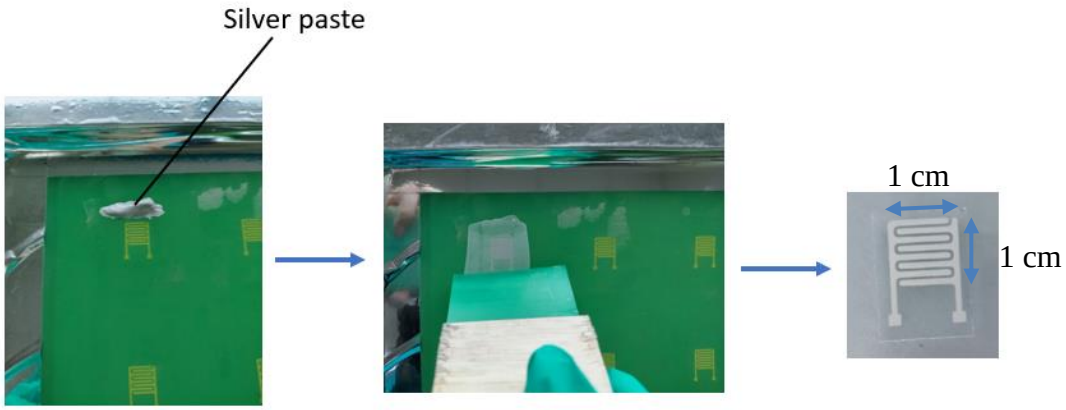


Figure 3.6: The screen-printing process to form electrode.

3.3.3 Sensing Material Deposition

3.3.3.1 Solution Preparation

Three types of solutions, which are graphene power with acetone, ethanol, and DI water are prepared. Before performing any measurements, ensure that the analytical balance is net weighted, as shown in Figure 3.7. After that, each solution is prepared one by one. For the first solution, 0.01 g of graphene is placed in a beaker and its mass is measured with the analytical balance, followed by 10 g of DI water being added to the same beaker. For preparing the second solution, the same quantity of graphene is added into another beaker along with 10 g of ethanol solvent. Repeat the same step by replacing 10 g of ethanol solvent with 10 g of acetone solvent. To reduce the evaporation of VOC solvent that is contained in the solution, parafilm is used to cover the whole top of the breaker. Later, all the solutions are sonicated for 30 minutes using an ultrasonic cleaner, as shown in Figure 3.8. The solutions that contain 0.02 g and 0.05 g of graphene power are prepared by applying the same steps as before. After that, all the previous steps are repeated by changing 10 g of DI water with 10 g of ethanol solvent and 10 g of acetone solvent. Each sonicated solution is illustrated in Figure 3.9, Figure 3.10, and Figure 3.11.

Figure 3.7: Net weight the analytical balance.

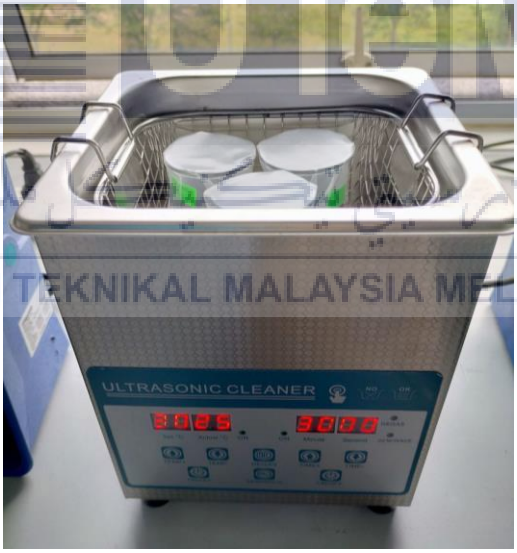


Figure 3.8: The solutions are sonicated with 30 minutes using ultrasonic cleaner.

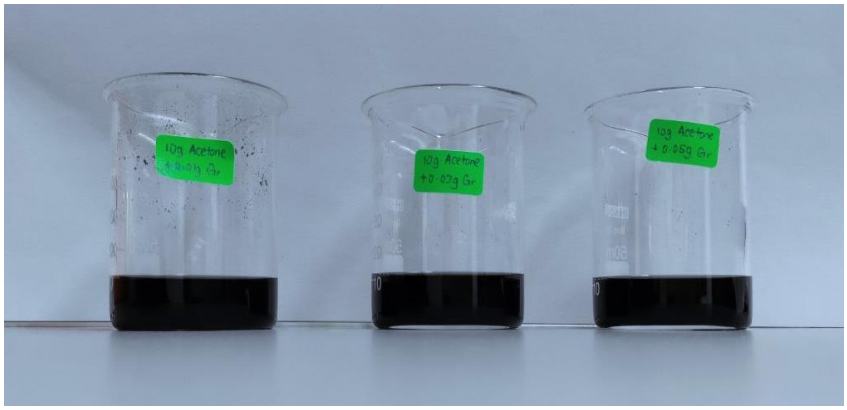


Figure 3.9: The sonicated solutions with 10 g acetone but with various amount of graphene which are 0.01 g of graphene, 0.02 g of graphene, and 0.05 g of graphene.

Figure 3.10: The sonicated solutions of 0.01 g, 0.02 g and 0.05 g graphene with 10 g of DI water arrange according on the picture.

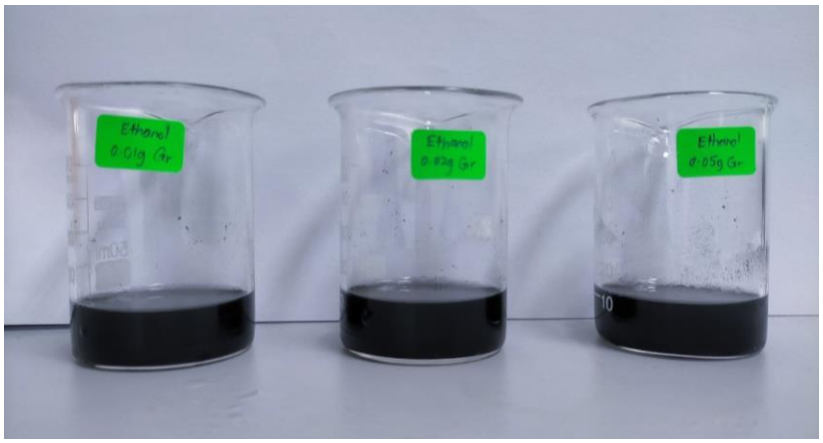


Figure 3.11: The sonicated solutions of 10 g ethanol with 0.01 g, 0.02 g, and 0.05 g of graphene arrange according from left to right.

3.3.3.2 Dropping Method on Substrate

In the dropping process, one drop of solution is dropped at the center of the electrode on substrates using a dropper as shown in Figure 3.12. The previous procedure is repeated using various sonicated solutions.

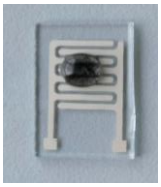


Figure 3.12: The substrates are dropped with the solution.

3.3.3.3 Annealing Process

The oven used (shown in Figure 3.13) is preheated for 150 ℃. The substrate that dropped with solution is undergo annealing process using oven at 150 ℃ for 10 minutes. This process is essential to ensure solution is evaporated and only crystalline graphene is left as a thin film.

Figure 3.13: The oven used in annealing process.

3.4 Current Voltage Characteristic

This session is important to check whether we have successfully fabricated a thin film gas sensor. After completing the fabricated thin film gas sensor, a desired voltage is set on LabVIEW 2010 software on a computer, and the computer is connected to a picoammeter to obtain the current. Then, the data obtained is used to plot the current-voltage graph (IV graph) with the Origin 2019b software. The resistance of the gas sensors can be found from the voltage and current obtained.

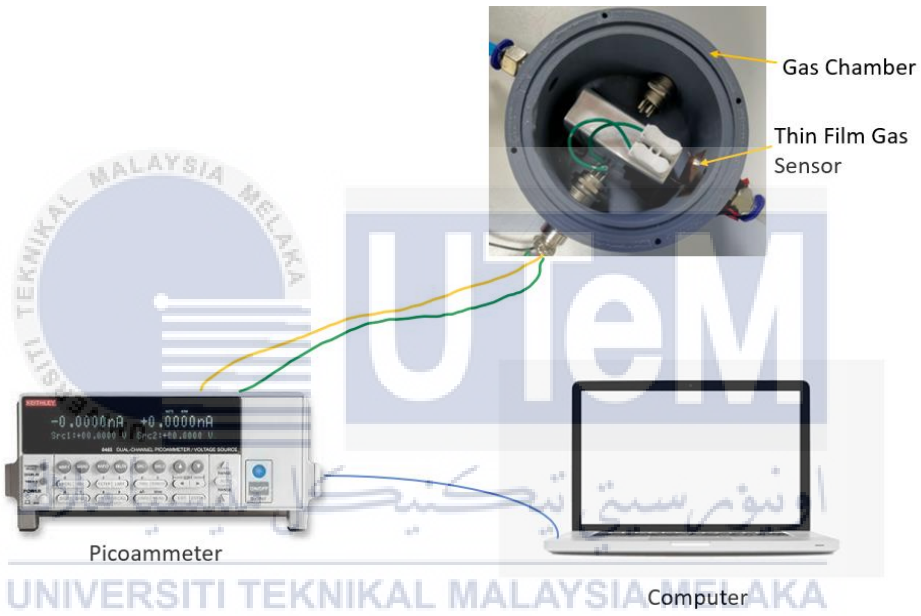


Figure 3.14: The connection of components during conducting IV measurement.

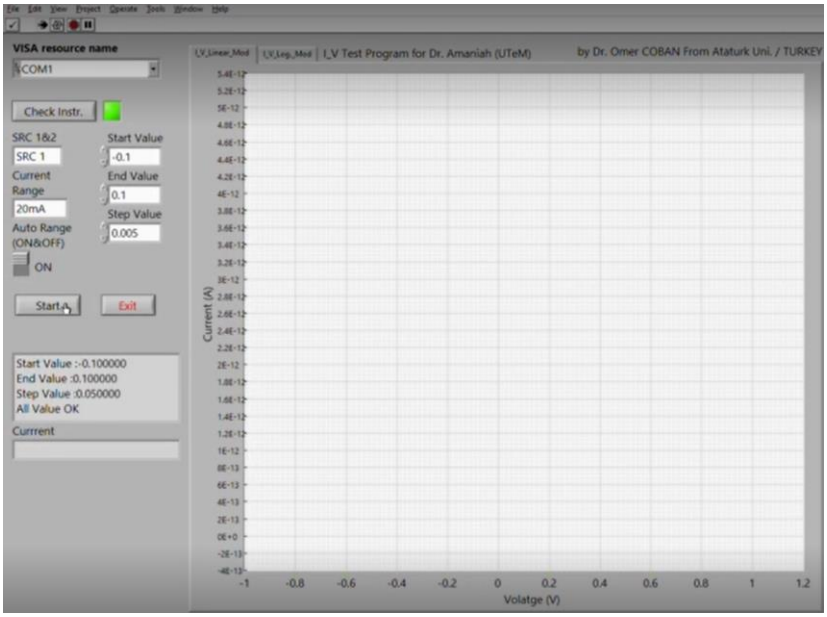


Figure 3.15: LabVIEW 2010 uses to obtain voltage-current measurement.

3.5 Setup of Gas Sensor Measurement

A solution that is used to produce VOC gas is prepared as shown in Figure 3.16. A magnetic stirrer bar is put into 100 ml of glassware glass washing first. Next, a different ratio of VOC solvent and DI water is added into the glassware. Then, the solution in the glassware is mixed with a magnetic stirrer at a speed of about 200 rpm for 5 minutes. Before starting to expose to gas, the solution in the glassware is put on the magnetic bar and heated for 30 minutes at the specific temperature according to Table 3.1 to produce VOC gas, but the glassware is not connected to the gas chamber. At first, the thin film gas sensor is placed inside the gas chamber facing the hose that will be connected to the glassware. The gas sensor is placed inside the gas chamber along with the current measurement process. The anode and cathode of the picoammeter are then connected to two wires from the gas chamber. The overall apparatus setup is shown in Figure 3.17. Next, the test gas journey begins. In this experiment, the desired voltage is supplied to the gas sensor using a picoammeter and

the setting is made using LabVIEW 2010 software (as shown in Figure 3.18). The current that passes through the thin film gas sensors at normal atmospheric pressure is measured using a picoammeter for 5 minutes of stabilization. Then, the solution that produces VOC gas in the glassware is connected to the end of the gas chamber that is directly facing the gas chamber for 5 minutes to obtain the response time. After 5 minutes, the hose from the glassware is disconnected from the gas chamber, and the recovery time is observed. All the data obtained from the LabVIEW 2010 software is transferred and plotted through the Origin 2019b software. The sensitivity of a gas sensor is identified by calculating the change of resistance values before and after the evaporation of VOC gases. The response time and recovery time can be obtained from the graph by using the methods as stated in Figure 2.13 and Figure 2.14 respectively.

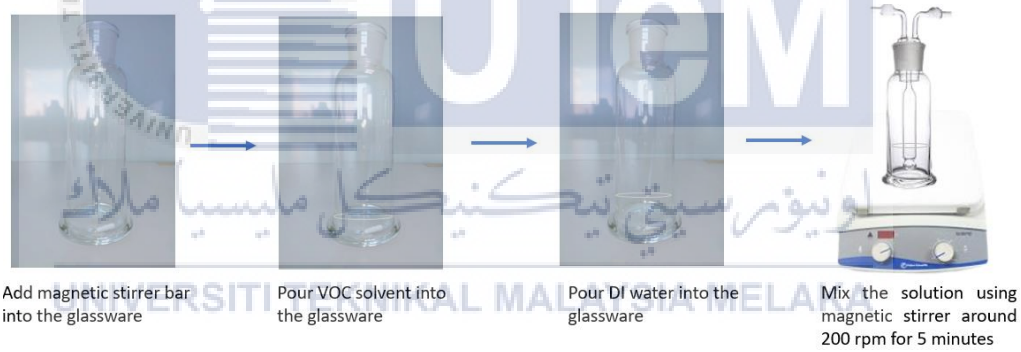


Figure 3.16: Preparing solution process.

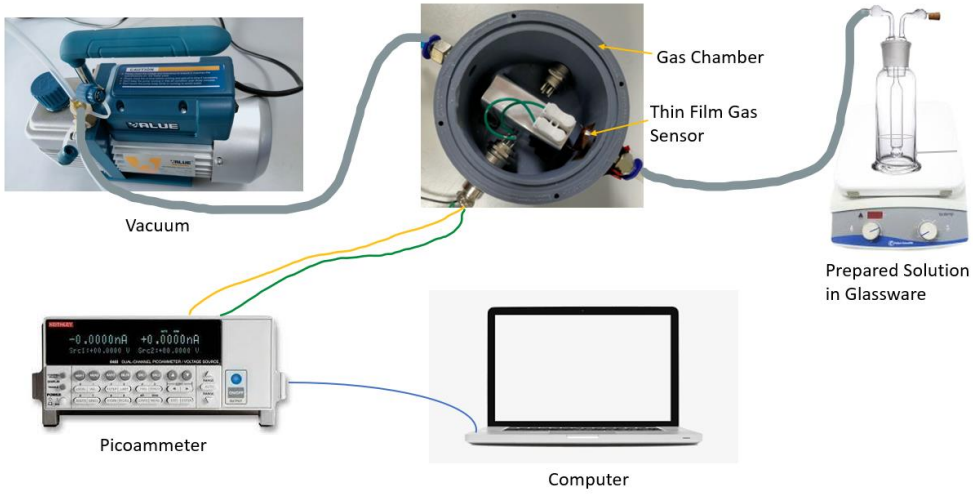


Figure 3.17: The setup apparatus before test VOC gas.

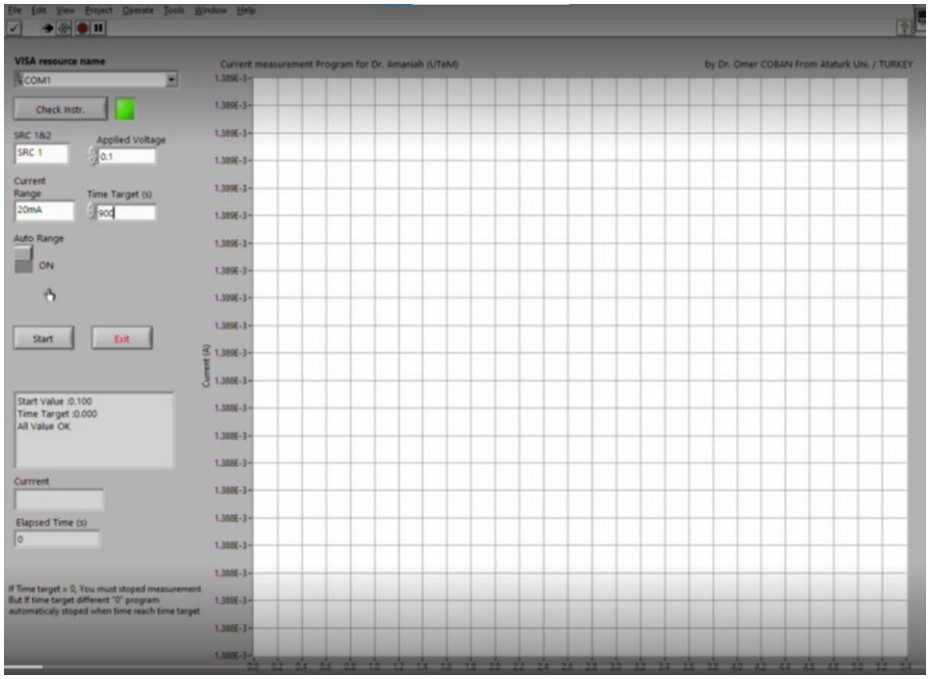


Figure 3.18: LabVIEW 2010 uses to obtain current measurement.

3.6 VOC Gas Measurement

3.6.1 Setting for VOC Gas Measurement

The targeted VOC gases to be tested in this project are acetone and ethanol. Each of them has a different boiling point, as shown in Table 3.1. The assumption of the boiling temperature for the solution to produce the VOC gases is made by finding the average range between the boiling point of the target VOC gas and DI water (whose boiling point is 100℃). The heating temperature that is used to evaporate the VOC gas is greater than the assumption of the boiling point temperature with respect to the mixed solution.

Table 3.1: The boiling point of the VOC gases and the assumption of heating temperature used in this project.

Type of VOC gas	Boiling point	Reference	Assumption of boiling point temperature for the mixed solution	Heating temperature to evaporate the VOC gas used in this project
Acetone	56.05℃	[38]	$\frac{100℃ - 56.05℃}{2} + 56.05℃ = 78.03℃$	90℃
Ethanol	78.37℃	[39]	$\frac{100℃ - 78.37℃}{2} + 78.37℃ = 89.19℃$	100℃

3.6.2 Calculation Amount of VOC Gases

Identify the amount of VOC gases evaporated from the solution as an important part. It helps us to identify the sensitivity, response time, and recovery time of the fabricated gas sensor toward the targeted VOC gases. The following are the steps to determine the amount of target VOC solvent and water required to produce the specific concentration of evaporated VOC vapour in ppm.

Assume 1 mg VOC solvent in 1 litre solution = 1 ppm, hence

$$\frac{x}{1\ \text{mg}} = \frac{\alpha}{1\ \text{ppm}}$$

where

x = The required mass of VOC solvent in mg to obtain desired concentration of VOC gases in mg.

α = The desired concentration of VOC gases in ppm.

Due to n% of denatured solvent is used, therefore

$$y = \frac{x}{\left(\frac{n}{100}\right)}$$

where

y = Mass of the n% of denatured solvent used in mg.

The density of the solvents is listed in Table 3.2.

Table 3.2: The density of the solvents depended on their heating temperature in Table 3.1.

Solvent	Temperature	Density	References
Acetone	90℃	706.9 kg/m^3= 0.7069 g/mL	[40]
Ethanol	100℃	713.6 kg/m^3= 0.7136 g/mL	[41]

Hence, the volume of the solvent required to produce α ppm can be identify from the density of the solvent used in Table 3.2.

$$v_{ml} = \frac{y}{\rho}$$

where

$v_{ml} =$ Volume of solvent in ml per 1 litre .

$y =$ Mass of solvent in g.

$\rho =$ Density of solvent in g/ml.

Due to 100 ml of solution is used in this experiment and compared with the assuming of a gas sensor in 1 litre. Therefore, the volume of the solvent that is obtained previously is multiplied by $\frac{100\text{ ml}}{1000\text{ ml}} = \frac{1}{10}$. Hence, the volume of solvent used in this experiment will be $v = \frac{1}{10} \times v_{ml}$.

From the above calculation and the content of the acetone and ethanol solvents used to produce VOCs vapour as in Figures 3.17 and 3.18, the information can be concluded as Table 3.3.

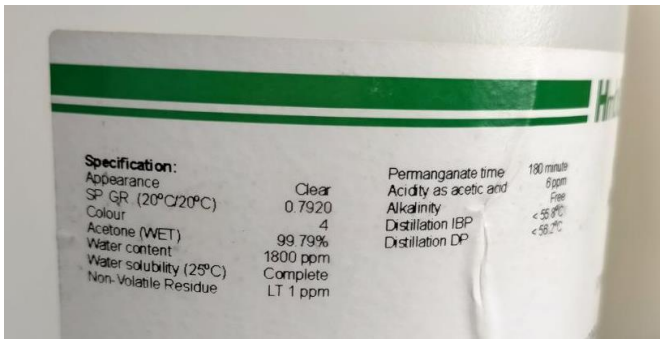


Figure 3.19: The content of acetone solvent.

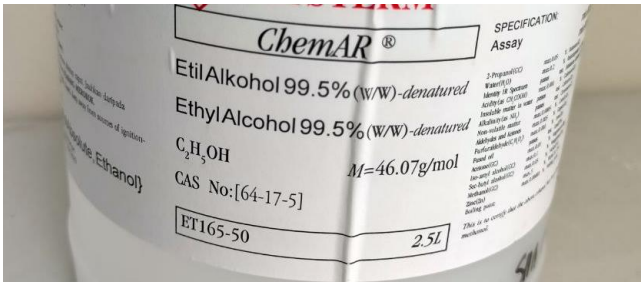


Figure 3.20: The information about the ethanol solvent used.

Table 3.3: The concentration of VOC gases required depend on their amount of solvent and DI water contain in the solution.

Type of VOCs gas produced	Type of solvent used	Amount of solvent	Amount of DI water	Concentration of VOC gases produced
Acetone vapour	99.79% Acetone	50 ml	50 ml	355.57×10^3 ppm
Ethanol vapour	99.5% Ethanol	50 ml	50 ml	355.02×10^3 ppm

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

In this session, the morphology of the thin films will be discussed using a scanning electron microscope (SEM). Besides, the current-voltage measurements are used to determine the resistances of the samples. The gas sensors are tested with two different types of VOC gas, which are acetone (the samples that are labelled with a “b” at the end) and ethanol (the samples that are labelled with a “a” at the end). Furthermore, the current measurements of the various thicknesses of thin film gas sensors that are fabricated with various solutions. Through the current measurement graph, the sensitivity, response time, and recovery time can be obtained.

4.2 Morphology of Thin Film

The structure of the gas sensors can be determined by using a scanning electron microscope (SEM) to magnify a thin film of gas sensor. Due to the fact that the structure of thin film on both types of substrates is the same, the samples using substrates of Kapton film are used to do SEM analysis. A sputtering technique is applied to obtain an ultra-thin gold coating on the thin film's surface before it undergoes magnification using SEM. This will increase the number of electrons that can be detected from the surface of the samples in the SEM. Figure 4.1 shows the structure of graphene power, whereas Figures 4.2, 4.3, and 4.4 show the structure of thin films at magnifications of 500 times (at the left of each figure) and 1000 times (at the right of each figure).

The graphene thin film structure dispersed on the substrate will affect the area that allows the gas to pass through the sensing material. If the thin film contains more gaps in the sensing material, it allows more gas to pass through it, so it can sense more gas at a time. Samples A2a, A5a, D1a, E1a, and E2a show many gaps in the sensing material even though the thin film of the samples is magnified 1000 times using SEM. Hence, more gaps allow more gas to diffuse into the thin films. Samples D2a, and D5a show fewer gaps in the thin film at both 500 and 1000 times magnification, indicating less gas can diffuse in the thin films.

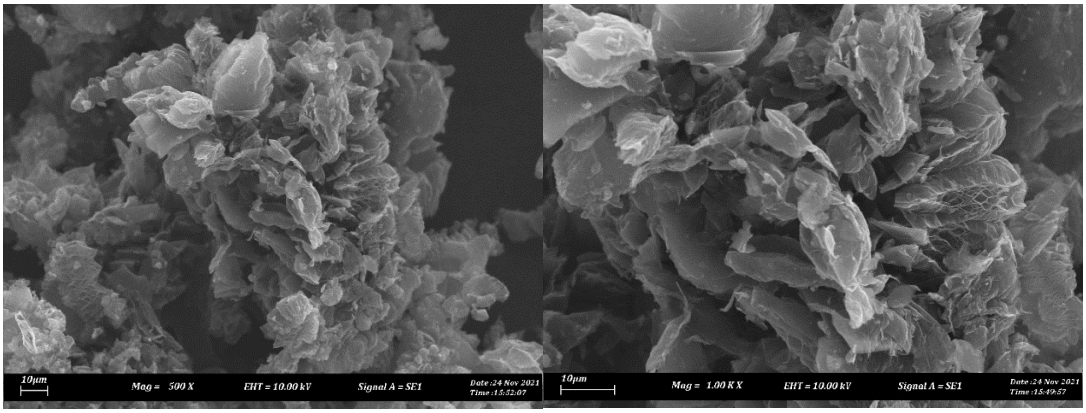


Figure 4.1: The magnification 500 and 1000 times of graphene power.

(a)

(b)

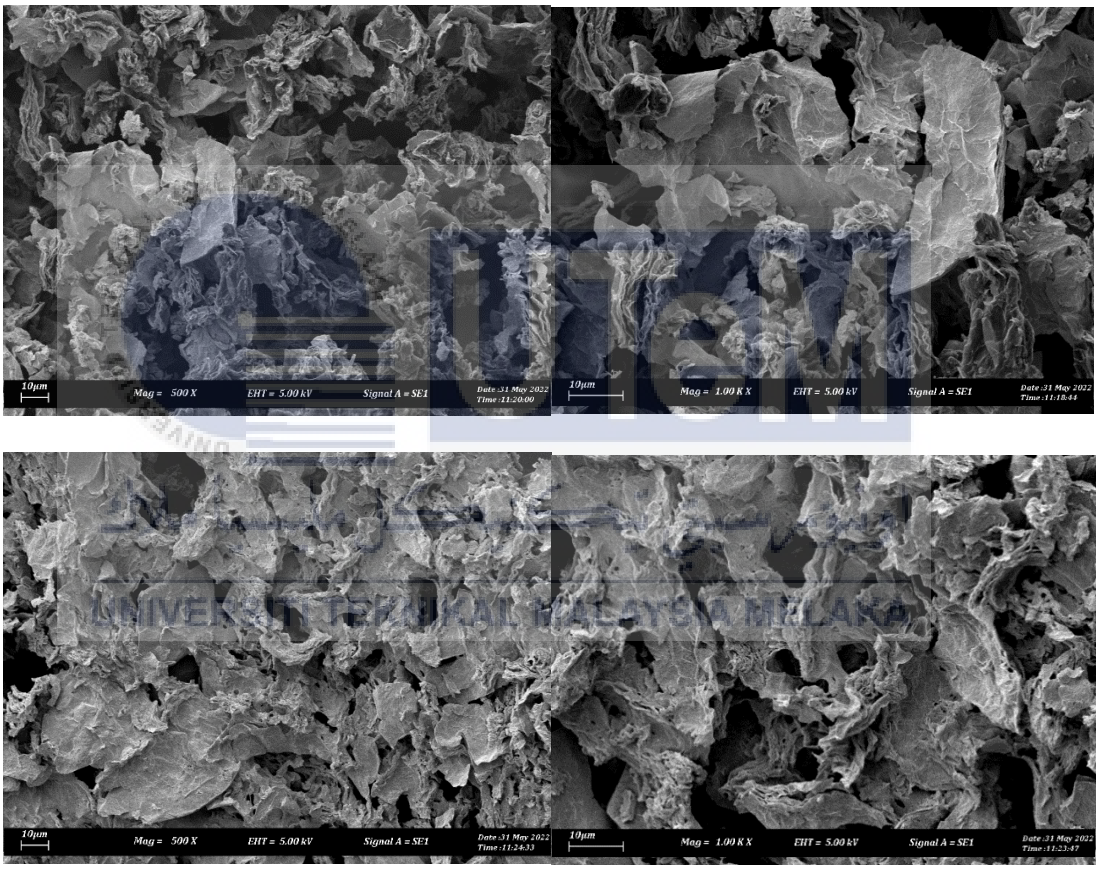


Figure 4.2: The magnification 500 and 1000 times of samples (a) A2a and (b) A5a respectively.

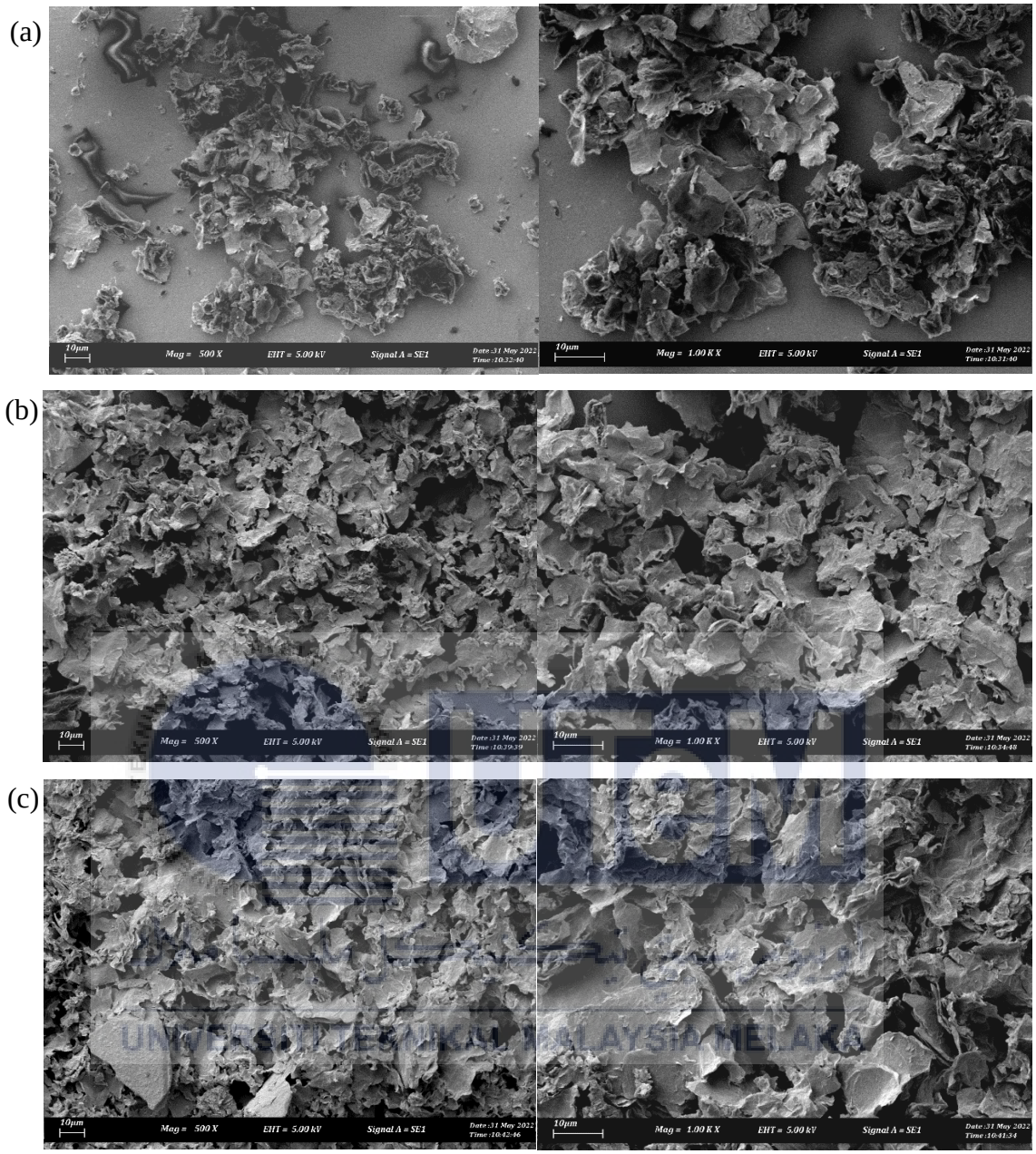
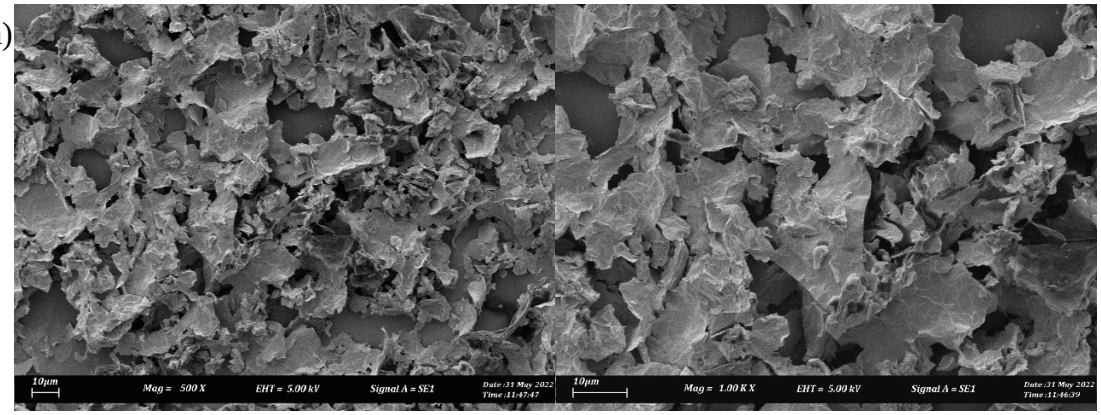


Figure 4.3: The magnification 500 and 1000 times of samples (a) D1a, (b) D2a and (c) D5a respectively.

(a)



(b)

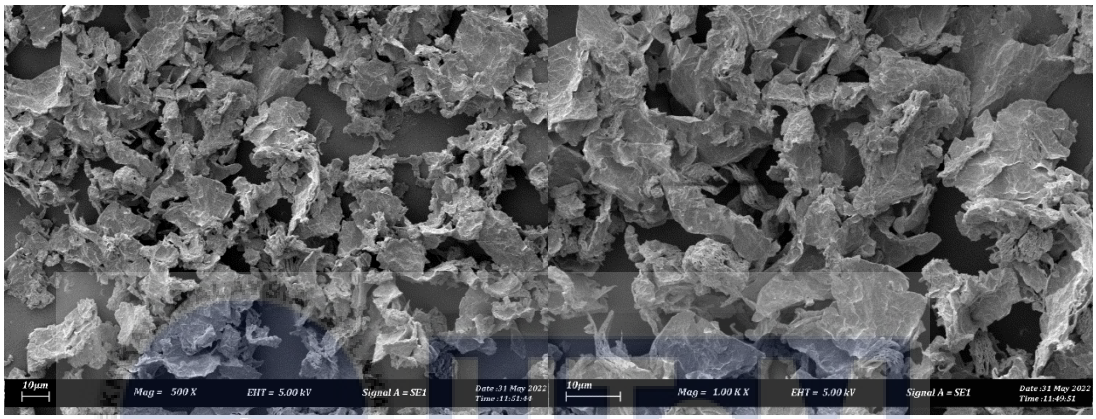


Figure 4.4: 500 and 1000 times magnification of samples (a) E1a, and (b) E5a respectively.

4.3 Fabrication of Gas Sensor

Table 4.1 shows the gas sensors are labelled based on the type of solution, substrate, and exposed gas. Although one drop of various solutions is applied to the substrates during the fabrication process, the thin film formed on the substrates varied slightly according to Figures 4.5, 4.6, 4.7, and 4.8. All the samples that are fabricated with the acetone solvent are widely dispersed on the substrates. They are the most adhesive compared to the samples that fabricated with ethanol and DI water. All samples that are fabricated with DI water are not adhesive, due to the solutions being deposited in a bunch on the substrates. The samples that are fabricated with ethanol solvent with 0.01 g of graphene power are dispersed widely on the substrates, whereas the thin films that are formed with ethanol solvent and 0.02 g and 0.05 g of graphene power are dispersed in a bunch. If the solutions are deposited in a bunch on the substrate, the sensing material will not stick to the substrate, causing the area of detection to be less than usual.

Table 4.1: The labelling of gas sensors depended on the type of solutions and substrates.

Type of solution	Samples that expose to ethanol gas						Samples that expose to acetone gas					
	0.01 g Gr		0.02 g Gr		0.05 g Gr		0.01 g Gr		0.02 g Gr		0.05 g Gr	
	Glass	Kapton film	Glass	Kapton film	Glass	Kapton film	Glass	Kapton film	Glass	Kapton film	Glass	Kapton film
Acetone	A-1a	A1a	A-2a	A2a	A-5a	A5a	A-1b	A1b	A-2b	A2b	A-5b	A5b
DI	D-1a	D1a	D-2a	D2a	D-5a	D5a	D-1b	D1b	D-2b	D2b	D-5b	D5b
Ethanol	E-1a	E1a	E-2a	E2a	E-5a	E5a	E-1b	E1b	E-2b	E2b	E-5b	E5b

(a) (b) (c) (d) (e) (f) (g) (h) (i)

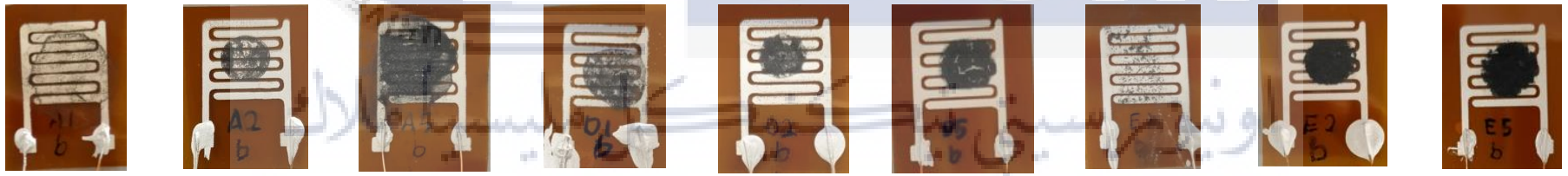
Figure 4.5: (a) A-1a, (b) A-2a, (c) A-5a, (d) D-1a, (e) D-2a, (f) D-5a, (g) E-1a, (h) E-2a, and (i) E-5a are the samples that will be used to expose to ethanol gas.

(a) (b) (c) (d) (e) (f) (g) (h) (i)

Figure 4.6: (a) A1a, (b) A2a, (c) A5a, (d) D1a, (e) D2a, (f) D5a, (g) E1a, (h) E2a, and (i) E5a are the samples that will be expose to ethanol gas.

(a) (b) (c) (d) (e) (f) (g) (h) (i)

Figure 4.7: (a) A-1b, (b) A-2b, (c) A-5b, (d) D-1b, (e) D-2b, (f) D-5b, (g) E-1b, (h) E-2b, and (i) E-5b are the samples that will expose acetone gas.



(a) (b) (c) (d) (e) (f) (g) (h) (i)

Figure 4.8: (a) A1b, (b) A2b, (c) A5b, (d) D1b, (e) D2b, (f) D5b, (g) E1b, (h) E2b, and (i) E5b are the samples that will used to expose acetone gas.

4.4 IV Characteristics

The samples undergo current-voltage measurement after they have been fabricated. The supply voltage from 0.1V up to 10V is applied to check the breakdown voltage of the gas sensors shown in Appendices D, E, F, and G. Most of the samples have a breakdown voltage of 10V due to the operation of the picoammeter being in the 10V range. Only the samples that are fabricated with 0.05 g of graphene power and ethanol, and 0.05 g of graphene power and DI water, have a breakdown voltage less than 10V, which are 0.6V and 0.2V respectively. Hence, 0.1V is used as the supply voltage due to this voltage being lower than the breakdown voltage of all samples.

0.1V of supply voltage is set using LabVIEW 2010 software to observe the change of current for each of the gas sensors. Figures 4.9 and 4.10 show IV characteristics for all the fabricated gas sensors according to the label stated in Table 4.1. Tables 4.2 and 4.3 show all the resistance of the fabricated gas sensors by applying Ohm's law, which is $R = \frac{V}{I} = \frac{1}{\left(\frac{I}{V}\right)} = \frac{1}{\text{slope of the graph}}$.

All the IV graphs of the gas sensor in Figures 4.9 and 4.10 show linear lines. Through the IV graphs, different solutions used will have different stiffness that will affect the resistance. Most of the samples fabricated with the highest amount of graphene power (0.05g) have the stiffest slope of IV graphs compared to the samples with 0.01 g and 0.02 g of graphene power, and vice versa. The stiffer the slope of the IV line, the less the resistance value of the samples due to the slope of the IV graph is inversely proportional to the resistance. Hence, most of the samples that contain 0.05 g of graphene power have low resistance.

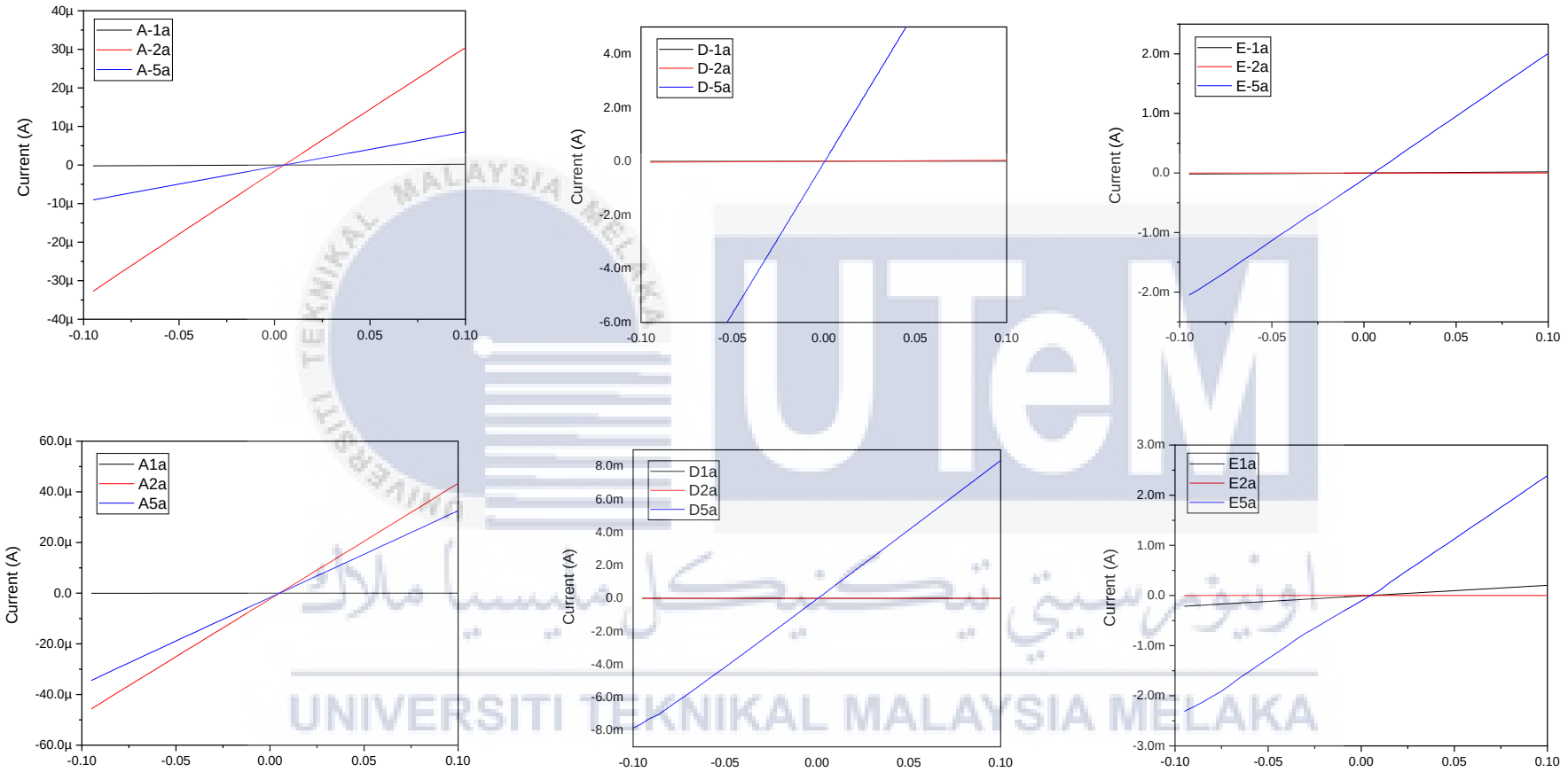


Figure 4.9: IV graph of the gas sensors use to test ethanol gas.

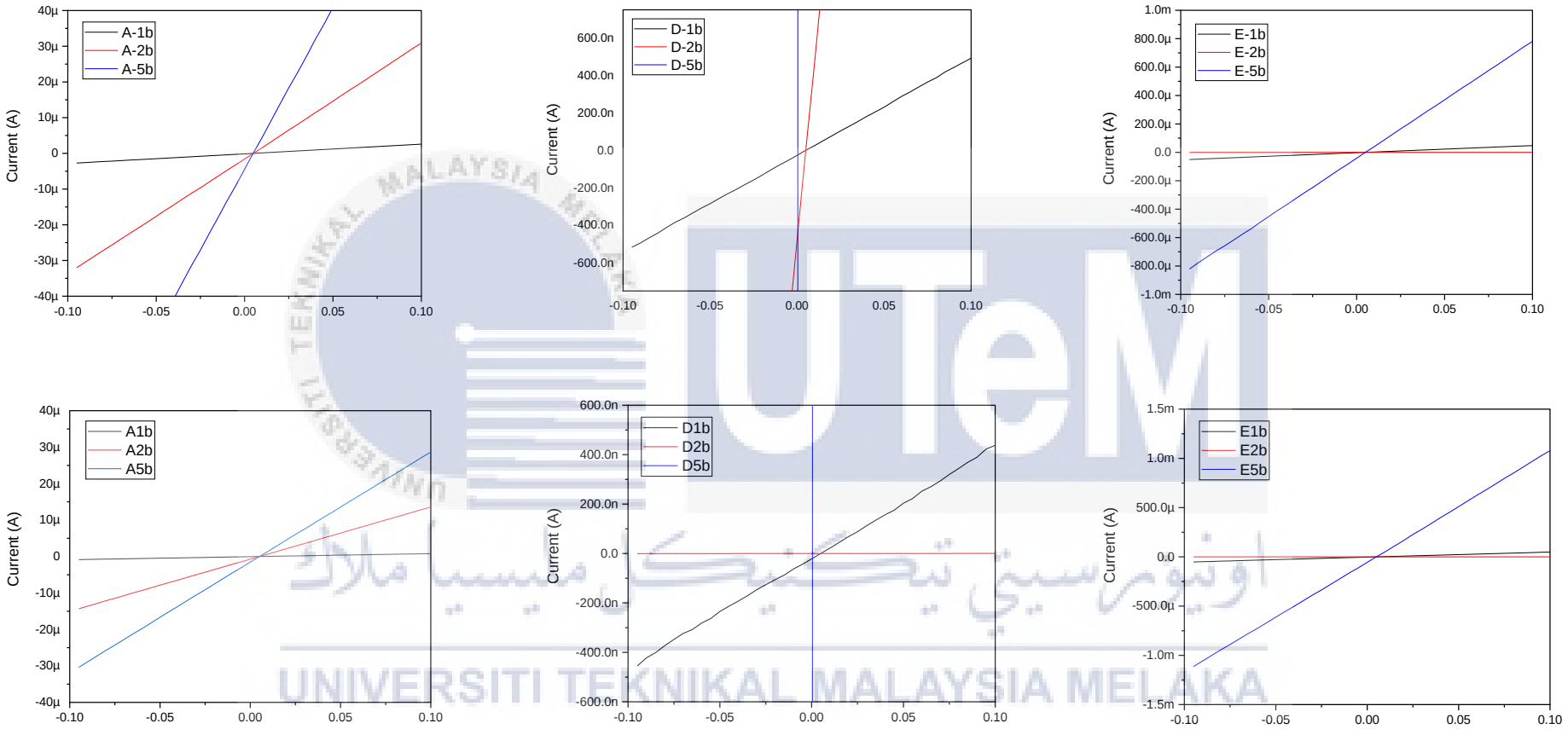


Figure 4.10: IV graph for gas sensors use to test acetone gas.

Table 4.2: The resistances of gas sensors that will be exposed to ethanol gas.

Sample	Slope of the graph according to Figure 4.9	Resistance	Sample	Slope of the graph according to Figure 4.9	Resistance
A-1a	2.10022×10^{-6}	476.14 kΩ	A1a	2.17942×10^{-7}	4.59 MΩ
A-2a	3.23634×10^{-4}	3.09 kΩ	A2a	4.56111×10^{-4}	2.19 kΩ
A-5a	9.03175×10^{-5}	11.07 kΩ	A5a	3.42994×10^{-4}	2.92 kΩ
D-1a	1.40125×10^{-5}	71.36 kΩ	D1a	2.39233×10^{-6}	418.00 kΩ
D-2a	3.72998×10^{-4}	2.68 kΩ	D2a	1.30583×10^{-6}	765.80 kΩ
D-5a	0.11211	8.92 Ω	D5a	0.08254	12.12 Ω
E-1a	2.03587×10^{-4}	4.91 kΩ	E1a	0.00214	467.29 Ω
E-2a	3.1968×10^{-9}	312.81 MΩ	E2a	2.8543×10^{-6}	350.35 kΩ
E-5a	0.02081	48.05 Ω	E5a	0.02415	41.41 Ω

Table 4.3: The resistances of gas sensors will be tested with acetone gas.

Sample	Slope of the graph according to Figure 4.10	Resistance	Sample	Slope of the graph according to Figure 4.10	Resistance
A-1b	2.70769×10^{-5}	378.18 Ω	A1b	8.38742×10^{-6}	119.23 kΩ
A-2b	3.2273×10^{-4}	3.10 kΩ	A2b	1.4303×10^{-4}	6.99 kΩ
A-5b	9.08542×10^{-4}	1.10 kΩ	A5b	3.02538×10^{-4}	3.31 kΩ
D-1b	5.17916×10^{-6}	193.08 kΩ	D1b	4.47976×10^{-6}	223.23 kΩ
D-2b	9.52572×10^{-5}	10.50 kΩ	D2b	3.10357×10^{-9}	322.21 MΩ
D-5b	0.10777	9.28 Ω	D5b	0.05895	16.96 Ω
E-1b	5.03987×10^{-4}	1.98 kΩ	E1b	5.14069×10^{-4}	1.95 kΩ
E-2b	3.0828×10^{-8}	32.44 MΩ	E2b	4.32251×10^{-9}	231.35 MΩ
E-5b	0.00822	121.65 Ω	E5b	0.01124	88.97 Ω

4.5 Current Measurement

For the first 5 minutes, it is used for stabilisation on the gas sensor. Then, the gas sensor is exposed to VOC gas for about 5 minutes, and response time is observed. The VOC gas is disconnected from the gas chamber, and the current of the gas sensor is measured for the next 5 minutes to obtain the recovery time. When a gas sensor is exposed to and discharged from VOC gas, its current will change when voltage is applied to it. Figures 4.11, 4.12, and 4.13 show the current measurement when the gas sensors are exposed to and discharged from ethanol gas, while Figures 4.14, 4.15, and 4.16 show the current changes of the gas sensors when exposed to and discharged from acetone gas. Tables 4.4 and 4.5 show the resistance before being exposed to gas, the sensitivity, response time, and recovery time of the gas sensor. The sensitivity of the gas sensor is found by using equation 2.1 in Chapter 2.7.1. The response time is obtained by finding the difference between 90% of the after-exposed gas that current becomes stable and 10% of the original reading, as shown in Figure 2.13, while the recovery time is determined by finding the difference in time between the 90% after-exposed gas reading and 10% of the original reading, as shown in Figure 2.14.

When the gas sensors are exposed to ethanol gas, the highest sensitivity of the gas sensors for each substrate is A-2a (0.30%) and D1a (3.24%). The shortest response time for the samples for each substrate is A-2a (244 s) and E1a (25 s). The recovery time for the samples for each substrate is A-2a (270 s) and E1a (28 s).

When the gas sensors are exposed to acetone gas, gas sensors A-5b (49.48%) and D1b (7.02%) have the highest sensitivity of the gas sensors for each substrate. Gas sensors A-2b (3 s) and E5b (1 s) have the shortest response time depending on each

substrate among all samples. Gas sensors D-5b (5 s) and D1b (165 s) have the shortest recovery time for each substrate.

The greater the difference in resistance before and after being exposed to gas, the higher the sensitivity of the gas sensor. For example, the sample A-5b has the highest sensitivity since the current change before and after being exposed to gas is the largest compared among all the samples by referring to the current measurement graph. As the value of the current change is larger, the change in resistance will also become larger.

The response time of a gas sensor depends on how fast the gas sensor can react with the gas and reach a stable state. For example, sample E5b takes the shortest time to reach a steady state after being exposed to gas. The better the gas sensor, the shorter the time taken to detect gas. The same concept applies to the recovery time of the gas sensor. The better the gas sensor, the less time the sample takes to discharge the gas. Sample D-5b takes the shortest time (5s) to discharge itself from gas.

Most of the gas sensors do not show the response to the gas because the graphs contain noise. This issue may be caused by the wires that are connected to both ends of the electrodes being folded many times during the connection. The high resistance of the gas sensors in MΩ such as samples E-2a, A1a, E-2b, and E2b show no response during expose to gas. This case may be due to fabricated electrodes being in an open circuit that allows very little current to pass through the gas sensor.

By referring to Tables 4.4 and 4.5, most of the gas sensors show more response to acetone gas than ethanol gas. Acetone is one type of ketone and ethanol is one type of organic compound. This is because acetone has electron-rich double bonds that make

it more reactive to react with the graphene thin film compared to ethanol, which consists of a single bond. Besides, the sample fabricated with the solution of 0.01g graphene and 10g DI water on Kapton film as substrate shows the high sensitivity to acetone and ethanol gas by observing Tables 4.4 and 4.5, where sample D1a has a sensitivity of 3.24% and sample D1b has a sensitivity of 7.02%. This is due to more gaps between graphene thin films deposited on Kapton film substrates (as shown in Figure 4.3(a)), allowing more gas to diffuse into the graphene thin film at the same time.

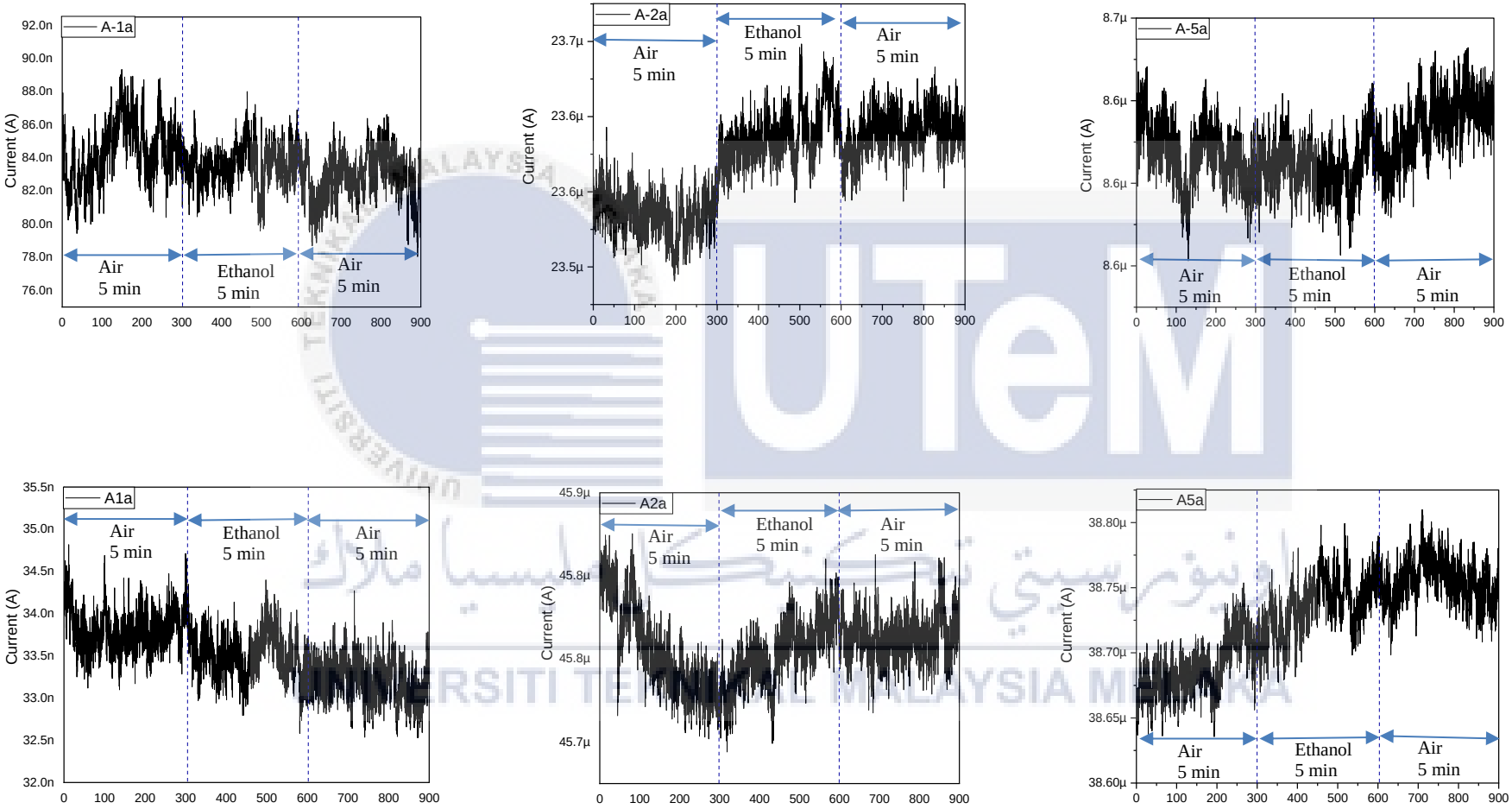


Figure 4.11: The current measurement graph for gas sensors that fabricated with acetone solution to expose ethanol vapour.

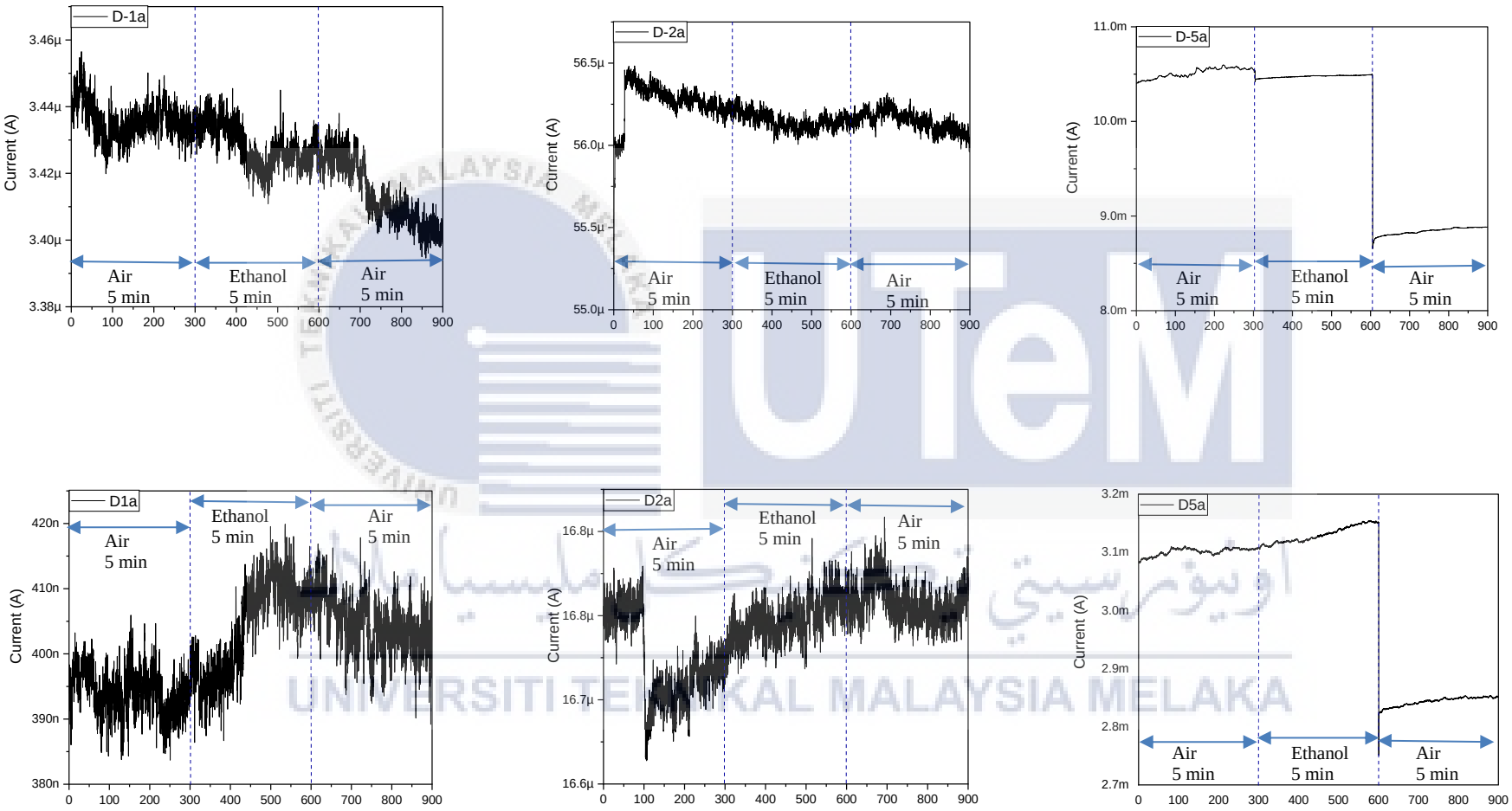


Figure 4.12: The current measurement graph for gas sensors that fabricated with distilled water solution to expose ethanol vapour.

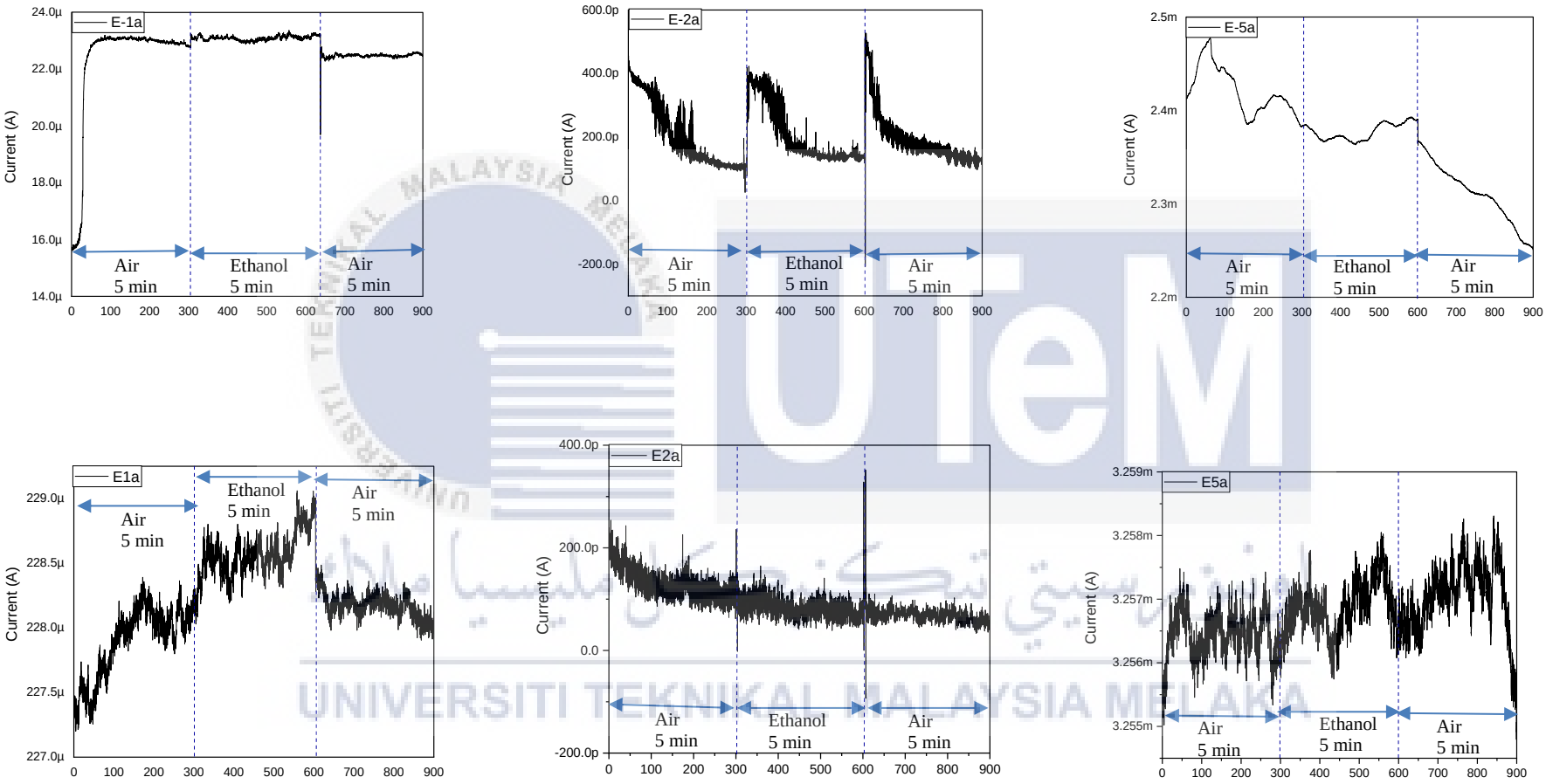


Figure 4.13: The current measurement graph for gas sensors that fabricated with ethanol solution to expose ethanol vapour.

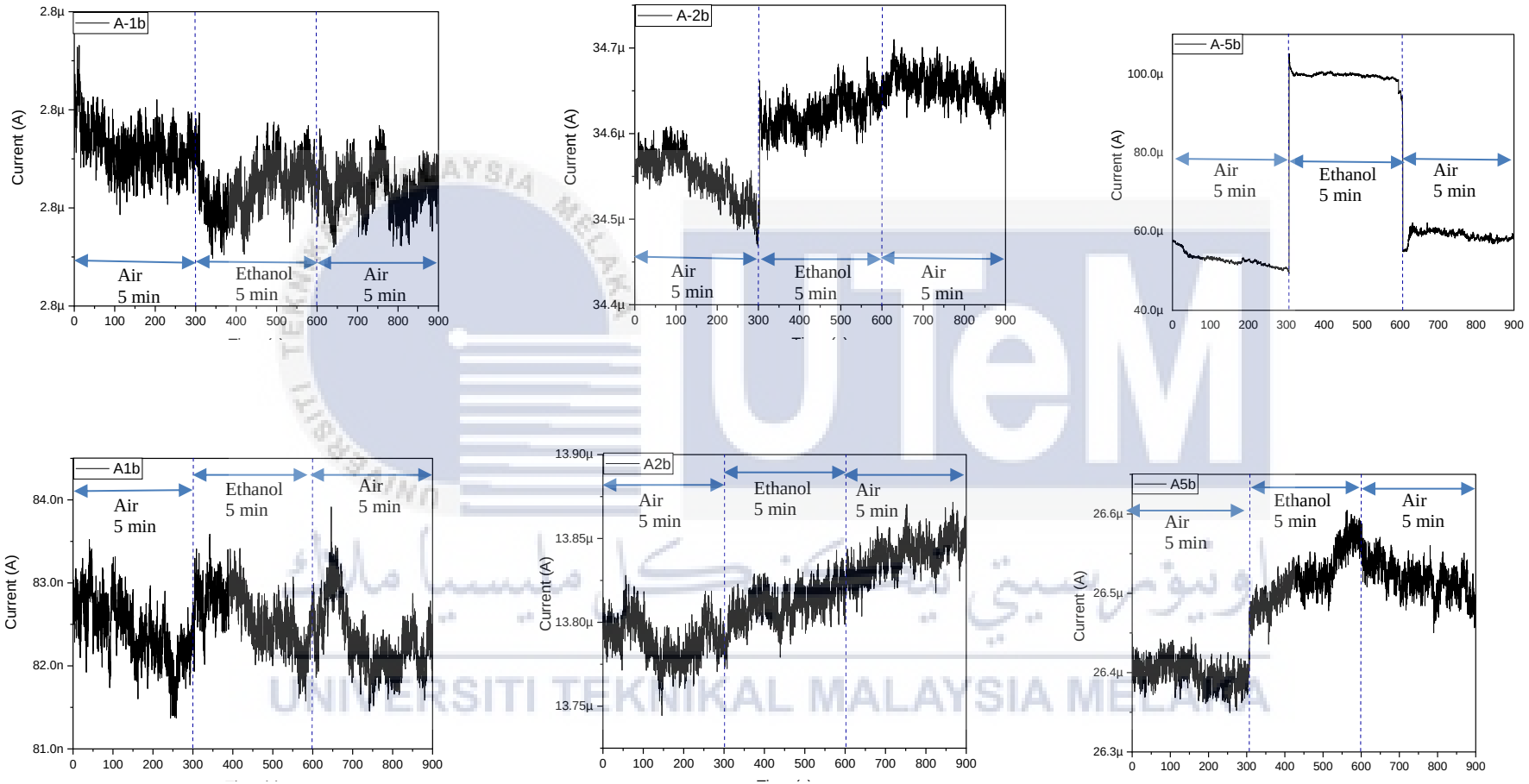


Figure 4.14: The current measurement graph for gas sensors that fabricated with acetone solution to expose acetone vapour.

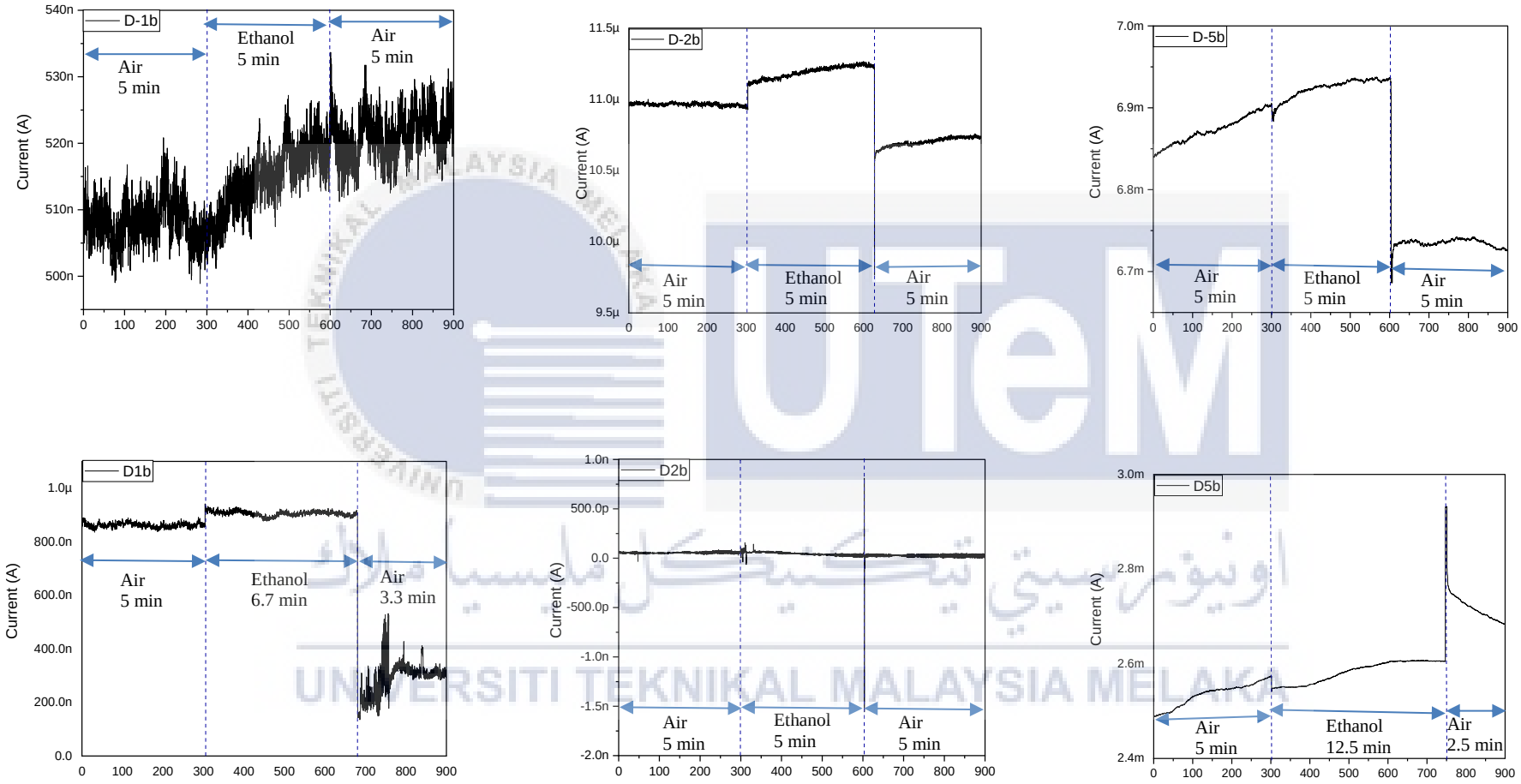


Figure 4.15: The current measurement graph for gas sensors that fabricated with distilled water solution to expose acetone vapour.

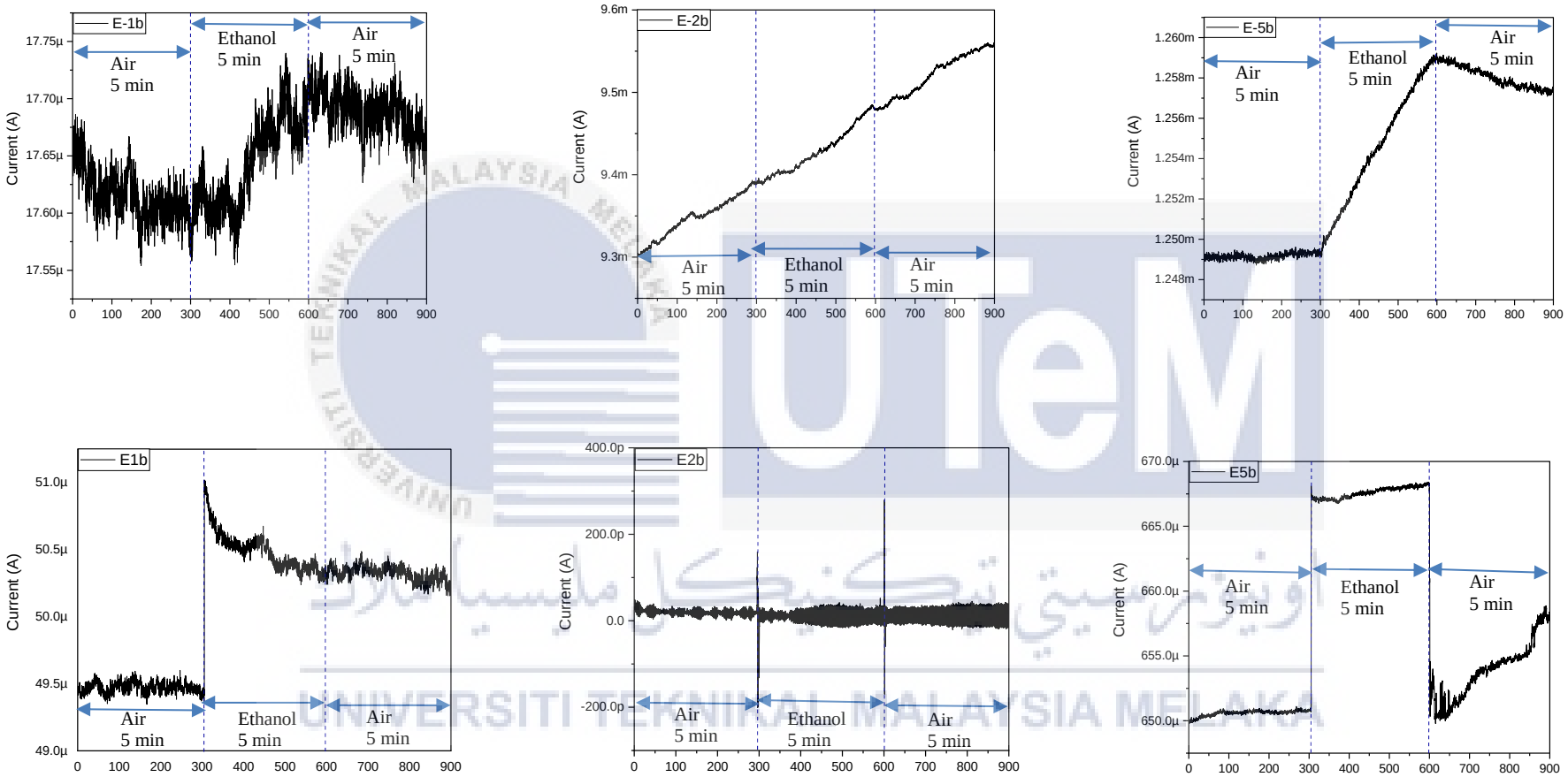


Figure 4.16: The current measurement graph for gas sensors that fabricated with ethanol solution to expose acetone vapour.

Table 4.4: The resistance, sensitivity, response time and recovery time for all the samples that expose to ethanol gas.

Sample	Resistance	Sensitivity	Response time	Recovery time	Sample	Resistance	Sensitivity	Response time	Recovery time
A-1a	476.14 kΩ	-	-	-	A1a	4.59 MΩ	-	-	-
A-2a	3.09 kΩ	0.30 %	244 s	270 s	A2a	2.19 kΩ	-	-	-
A-5a	11.07 kΩ	-	-	-	A5a	2.92 kΩ	0.14 %	260 s	270 s
D-1a	71.36 kΩ	-	-	-	D1a	418.00 kΩ	3.24 %	145 s	270 s
D-2a	2.68 kΩ	-	-	-	D2a	765.80 kΩ	-	-	-
D-5a	8.92 Ω	-	-	-	D5a	12.12 Ω	-	-	-
E-1a	4.91 kΩ	-	-	-	E1a	467.29 Ω	0.16 %	25 s	28 s
E-2a	312.81 MΩ	-	-	-	E2a	350.35 kΩ	-	-	-
E-5a	48.05 Ω	-	-	-	E5a	41.41 Ω	-	-	-

Table 4.5: The resistance, sensitivity, response time and recovery time of the samples that expose to acetone gas.

Sample	Resistance	Sensitivity	Response time	Recovery time	Sample	Resistance	Sensitivity	Response time	Recovery time
A-1b	378.18 Ω	-	-	-	A1b	119.23 kΩ	-	-	-
A-2b	3.10 kΩ	0.34 %	3 s	270 s	A2b	6.99 kΩ	0.25 %	260 s	205 s
A-5b	1.10 kΩ	49.48 %	15 s	45 s	A5b	3.31 kΩ	0.64 %	225 s	219 s
D-1b	193.08 kΩ	3.24 %	285 s	270 s	D1b	223.23 kΩ	7.02 %	5 s	165 s
D-2b	10.50 kΩ	1.60 %	4 s	50 s	D2b	322.21 MΩ	-	-	-
D-5b	9.28 Ω	0.47 %	230s	5 s	D5b	16.96 Ω	-	-	-
E-1b	1.98 kΩ	0.54 %	235 s	235 s	E1b	1.95 kΩ	1.78 %	69 s	270 s
E-2b	32.44 MΩ	-	-	-	E2b	231.35 MΩ	-	-	-
E-5b	121.65 Ω	0.77 %	260 s	270 s	E5b	88.97 Ω	2.43 %	1 s	270 s

CHAPTER 5

CONCLUSION

5.1 Conclusion

Among the three types of solutions used in this project, the solution with acetone solvent produces a well dispersed thin film on the substrate, although the amount of graphene power has been increased. Samples A-2a (3.09 kΩ), A2a (2.19 kΩ), A-5b (1.1 kΩ) and A5b (3.31 kΩ) have low resistances among gas sensors using acetone solvent for each substrate. D-5a (8.92 kΩ), D5a (12.12 Ω), D-5b (9.28Ω) and D5b (16.96 Ω) have low resistances throughout the samples with DI water on different substrates. E-5a (48.05 Ω), E5a (41.41 Ω), E-5b (121.65 Ω) and E5b (88.97 Ω) have

low resistance among the gas sensors that contain ethanol solvent on different substrates.

In this part, we will discuss the gas sensors when exposed to ethanol gas. The highest sensitivity of a gas sensor with a glass substrate is A-5b (49.48%), while for Kapton film is D1b (7.02%). The fastest response time of samples using glass is A-2a (244 s), while using Kapton film is E1a (25 s). The samples' fastest recovery time with glass is A-2a (270 s), while for Kapton film is E1a (28 s).

This paragraph will discuss the gas sensor when exposed to acetone gas. The highest sensitivity of a gas sensor with a glass substrate is A-5b (49.48%), while for Kapton film is D1b (7.02%). The gas sensor with the fastest time of response with a glass substrate is A-2b (3 s), while with Kapton film is E5b (1 s). The fastest recovery time of the gas sensor using glass is D-5b (5 s), while for Kapton film is D1b (165 s).

Graphene thin film gas sensors show more sensitive toward acetone gas than ethanol gas due to a more significant current change among the samples when exposed to acetone. The substrate that is suitable to detect acetone gas is glass, while for ethanol gas is Kapton film. Samples that contain 0.01g graphene with 10g DI water on Kapton film show the high sensitivity to acetone and ethanol gas, where sample D1a has a sensitivity of 3.24% and sample D1b has a sensitivity of 7.02%.

Because the thin film gas sensor is thin and small, it can be used to detect VOC gas leakage at a small surface area compared to the gas sensors sold in the market. Besides, it is suitable for people who are often exposed to VOC gas. By detecting the presence of VOC gas in the environment, we can take future action to prevent unpleasant incidents from happening.

5.2 Future Recommendation

The project that is related to the thickness of thin film gas sensor with the same method has not been discovered by researchers. Hence, the sensitivity, response time, and recovery time of most of the fabricated thin film gas sensors are not sufficiently high, and many things still need to be improved during the fabrication process.

The solution used to deposit on the substrate to form a thin film is less sticky on the substrate. It will make the thin film easy to remove from the substrate because the thin film gas sensor cannot function well. To improve the adhesion of sensing material on the substrates, the solvent used in fabricating thin films can be changed to a more adhesive solvent such as terpineol or polymer.

Besides, the amount of solution used to deposit on the substrate can be increased from one drop to two drops while maintaining the thickness of thin film below 1μm. This is to ensure the sensing material fully covers the surface area of the electrode to detect as much VOC gas as possible.

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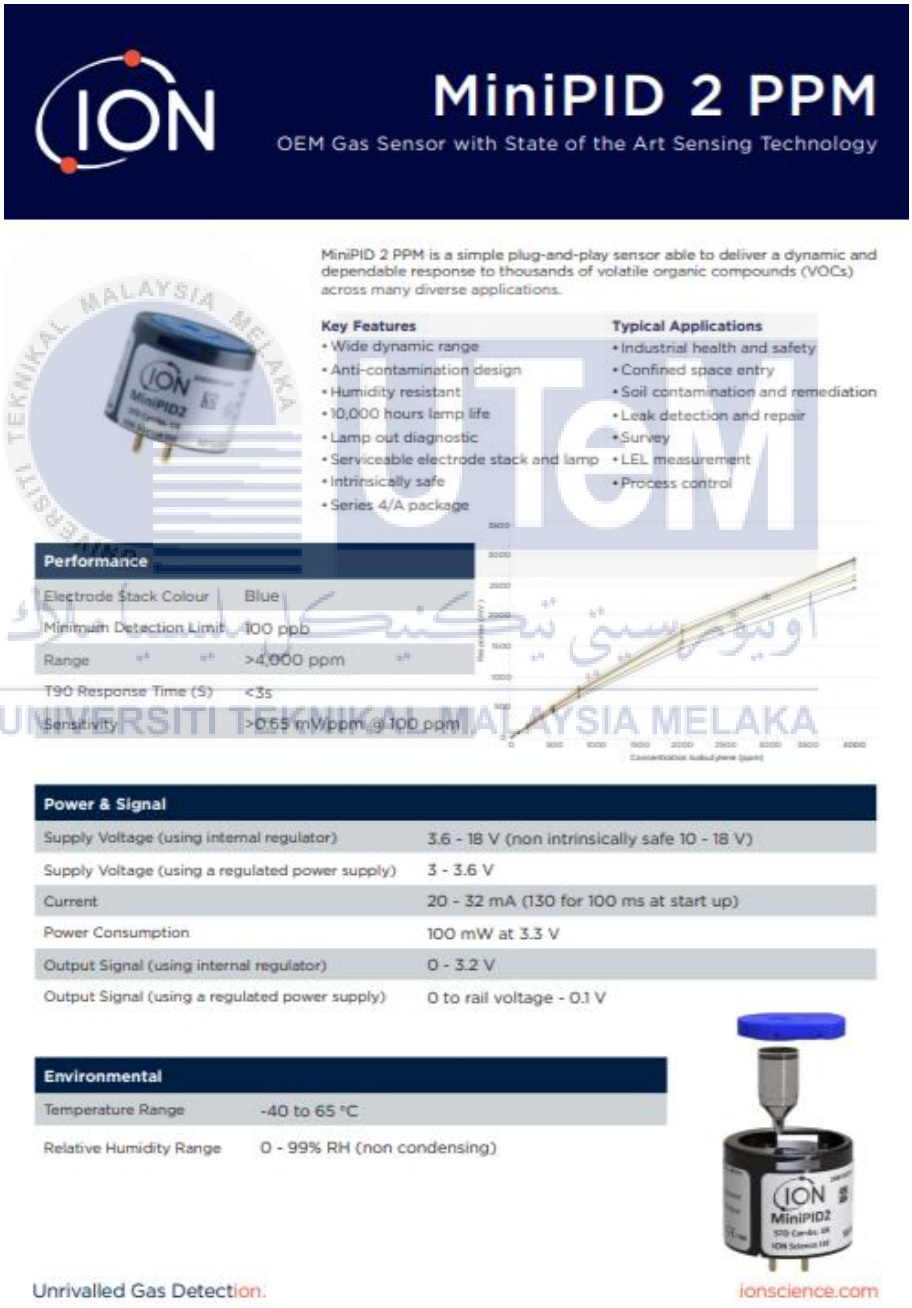
APPENDICES



MiniPID 2 PPM

OEM Gas Sensor with State of the Art Sensing Technology

MiniPID 2 PPM is a simple plug-and-play sensor able to deliver a dynamic and dependable response to thousands of volatile organic compounds (VOCs) across many diverse applications.

Key Features
- Wide dynamic range
- Anti-contamination design
- Humidity resistant
- 10,000 hours lamp life
- Lamp out diagnostic
- Serviceable electrode stack and lamp
- Intrinsically safe
- Series 4/A package

Typical Applications
- Industrial health and safety
- Confined space entry
- Soil contamination and remediation
- Leak detection and repair
- Survey
- LEL measurement
- Process control

Performance	
Electrode Stack Colour	Blue
Minimum Detection Limit	100 ppb
Range	>4,000 ppm
T90 Response Time (S)	<3s
Sensitivity	>0.65 mV/ppm @ 100 ppm

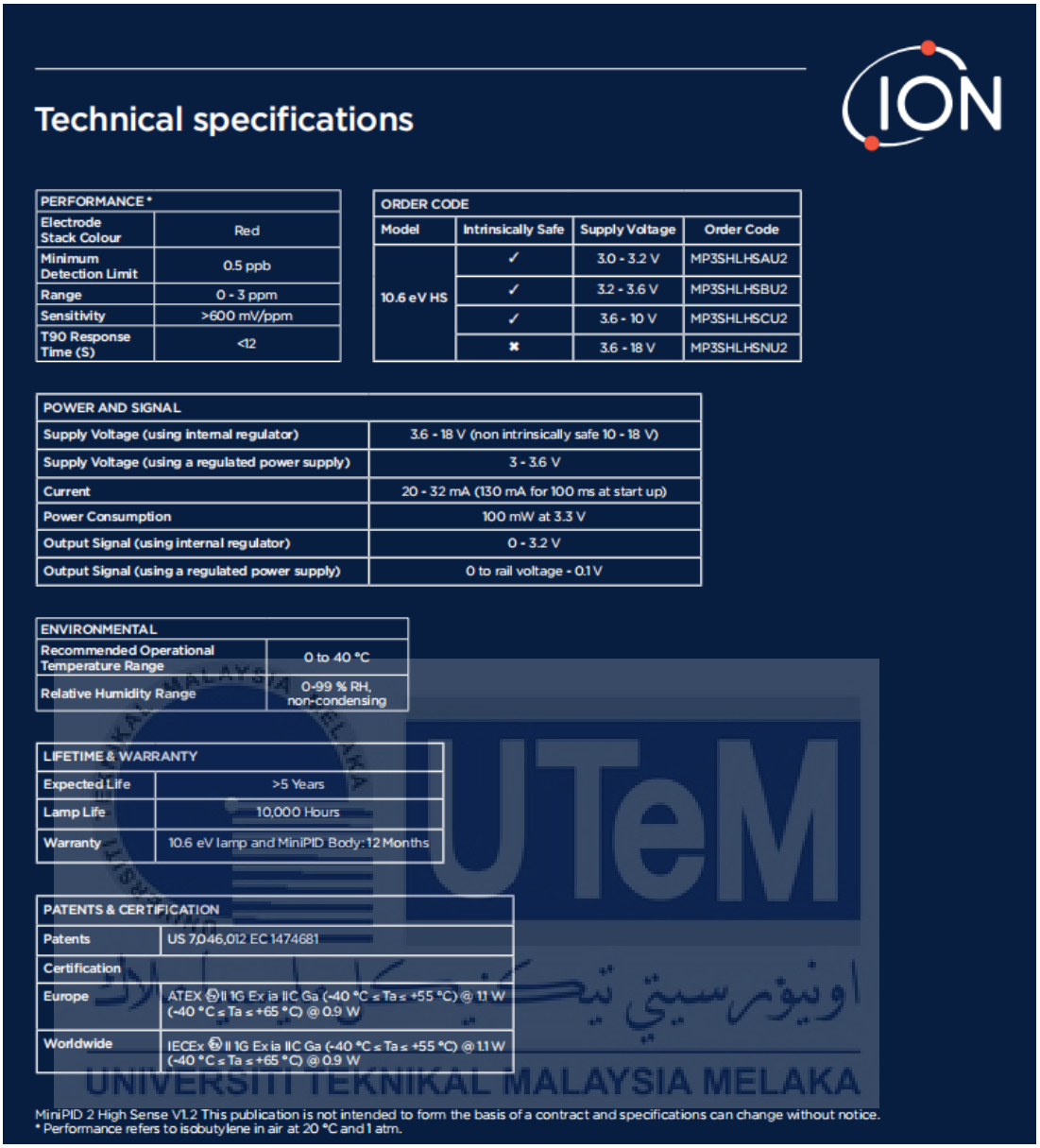
Power & Signal	
Supply Voltage (using internal regulator)	3.6 - 18 V (non intrinsically safe 10 - 18 V)
Supply Voltage (using a regulated power supply)	3 - 3.6 V
Current	20 - 32 mA (130 for 100 ms at start up)
Power Consumption	100 mW at 3.3 V
Output Signal (using internal regulator)	0 - 3.2 V
Output Signal (using a regulated power supply)	0 to rail voltage - 0.1 V

Environmental	
Temperature Range	-40 to 65 °C
Relative Humidity Range	0 - 99% RH (non condensing)

Unrivalled Gas Detection.

ionscience.com

Appendix A: Datasheet for MiniPID 2 PPM.



Technical specifications

PERFORMANCE *	
Electrode Stack Colour	Red
Minimum Detection Limit	0.5 ppb
Range	0 - 3 ppm
Sensitivity	>600 mV/ppm
T90 Response Time (S)	<12

ORDER CODE			
Model	Intrinsically Safe	Supply Voltage	Order Code
10.6 eV HS	✓	3.0 - 3.2 V	MP3SHLHSAU2
	✓	3.2 - 3.6 V	MP3SHLHSBU2
	✓	3.6 - 10 V	MP3SHLHSCU2
	✖	3.6 - 18 V	MP3SHLHSNU2

POWER AND SIGNAL	
Supply Voltage (using internal regulator)	3.6 - 18 V (non intrinsically safe 10 - 18 V)
Supply Voltage (using a regulated power supply)	3 - 3.6 V
Current	20 - 32 mA (130 mA for 100 ms at start up)
Power Consumption	100 mW at 3.3 V
Output Signal (using internal regulator)	0 - 3.2 V
Output Signal (using a regulated power supply)	0 to rail voltage - 0.1 V

ENVIRONMENTAL	
Recommended Operational Temperature Range	0 to 40 °C
Relative Humidity Range	0-99 % RH, non-condensing

LIFETIME & WARRANTY	
Expected Life	>5 Years
Lamp Life	10,000 Hours
Warranty	10.6 eV lamp and MiniPID Body: 12 Months

PATENTS & CERTIFICATION	
Patents	US 7,046,012 EC 1474681
Certification	
Europe	ATEX Ⓔ II 1G Ex ia IIC Ga (-40 °C ≤ Ta ≤ +55 °C) @ 1.1 W (-40 °C ≤ Ta ≤ +65 °C) @ 0.9 W
Worldwide	IECEx Ⓔ II 1G Ex ia IIC Ga (-40 °C ≤ Ta ≤ +55 °C) @ 1.1 W (-40 °C ≤ Ta ≤ +65 °C) @ 0.9 W

MiniPID 2 High Sense V1.2 This publication is not intended to form the basis of a contract and specifications can change without notice.
* Performance refers to isobutylene in air at 20 °C and 1 atm.

Appendix B: Datasheet for MiniPID 2 HS.

Technical specifications

Sensor
- Photoionization detector

Falco detection ranges and sensitivity with 10.6eV lamp fitted*
- 10.0 ppm, 0.001 ppm
- 50.0 ppm, 0.01 ppm
- 1000 ppm, 0.1 ppm
- 3000 ppm, 1 ppm

Falco TAC detection ranges and sensitivity with 10.0eV lamp fitted*
- 50.0 ppm, 0.01 ppm

Response time (T90)
- Diffused models: <30 seconds**

Accuracy
- ± 5% or ± 1 digit

User interface
- OLED high contrast white on black: 128 x 64 pixels
- Screen size: 35 mm (w) x 17.5 mm (h)
- 5 magnetic switches with LED confirmation (up, down, left, right & enter).

Status indicator
- Bright visible status indicator: RED, AMBER, GREEN

Output
- 4 - 20 mA
- 2 programmable relays
- RS 485 Modbus

Falco diffused environmental specification
- Operational temperature: -40 °C to 50 °C 0-100% RH and condensing humidity
- Storage temperature: -40 °C to 60 °C

Ingress Protection
- Main unit: IP65
- Sensor head: IP65

Power
- Working voltage: 12 to 40 Vdc
- Typical 2 W, Max. 7 watts

Mechanical interface
- 2 x cable entry points with 3/4" NPT threads (left and right)

Mounting points
- 2 x M8

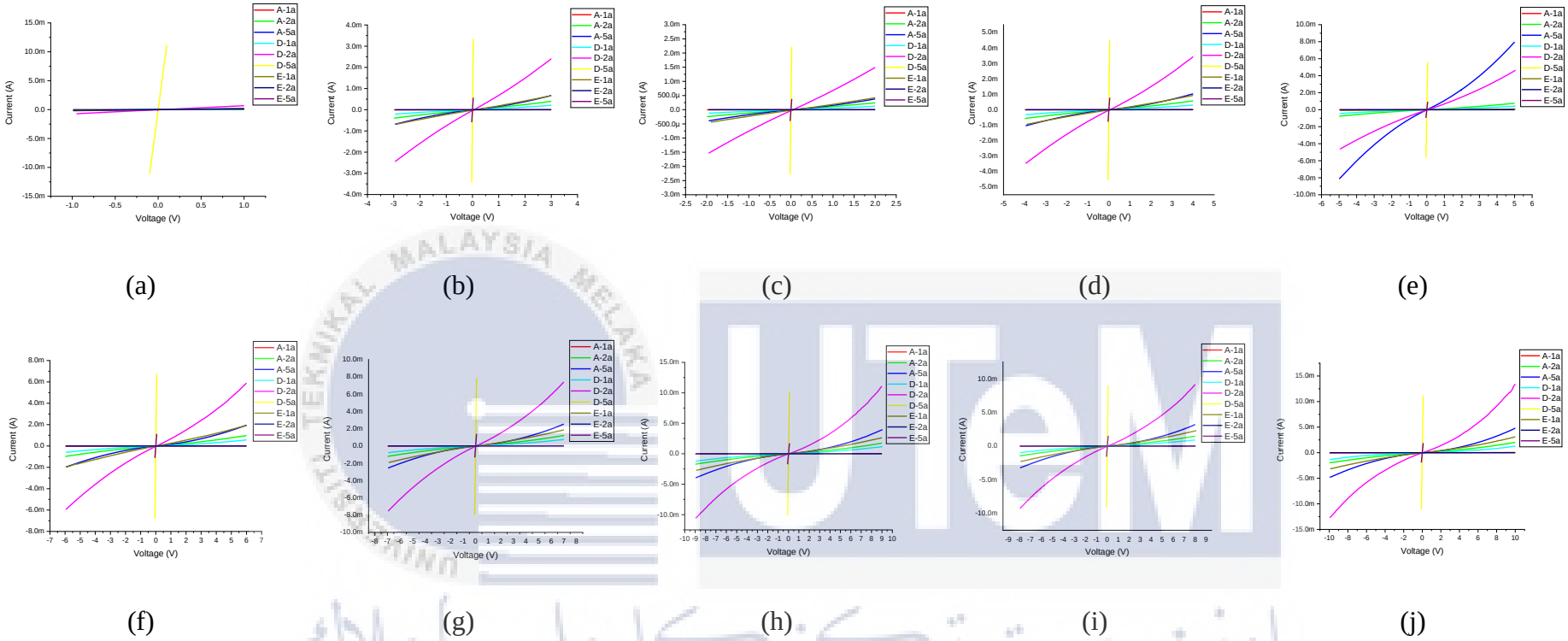
Weight & Dimensions
- 2.9 kg
- 205 (h) x 180 (w) x 125 (d) mm

EMC
- EMC Directive 2014/30/EU

Certification
- Ex II 2G db ib IIC T4 Gb
- Class 1, Div 1 Groups ABCD T4
- ISO9001:2015

* All specifications quoted are at calibration point and under the same ambient conditions.
Specifications are based on isobutylene calibration at 20 °C and 1000 mBar.
** When set to continuous measurement
Modbus® is a registered trademark of Schneider Electric
FALCO V1.9 This publication is not intended to form the basis of a contract and specification can change without notice.

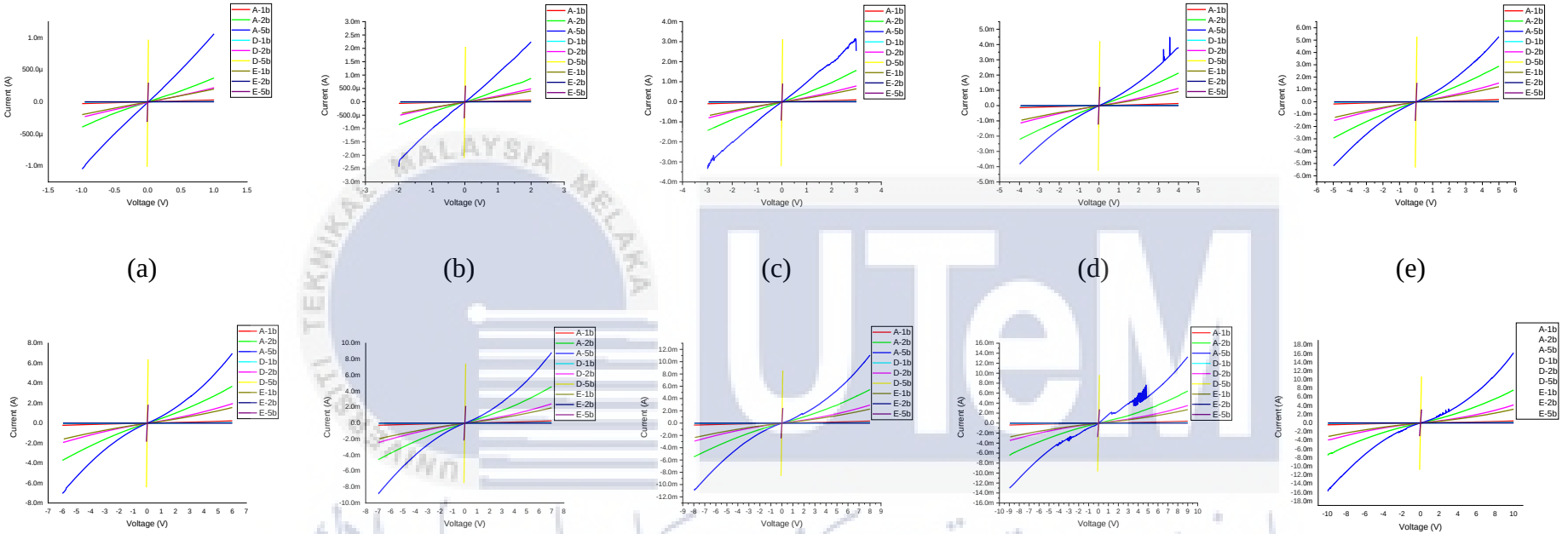
Appendix C: Datasheet for Falco pumped fixed VOC gas monitor.



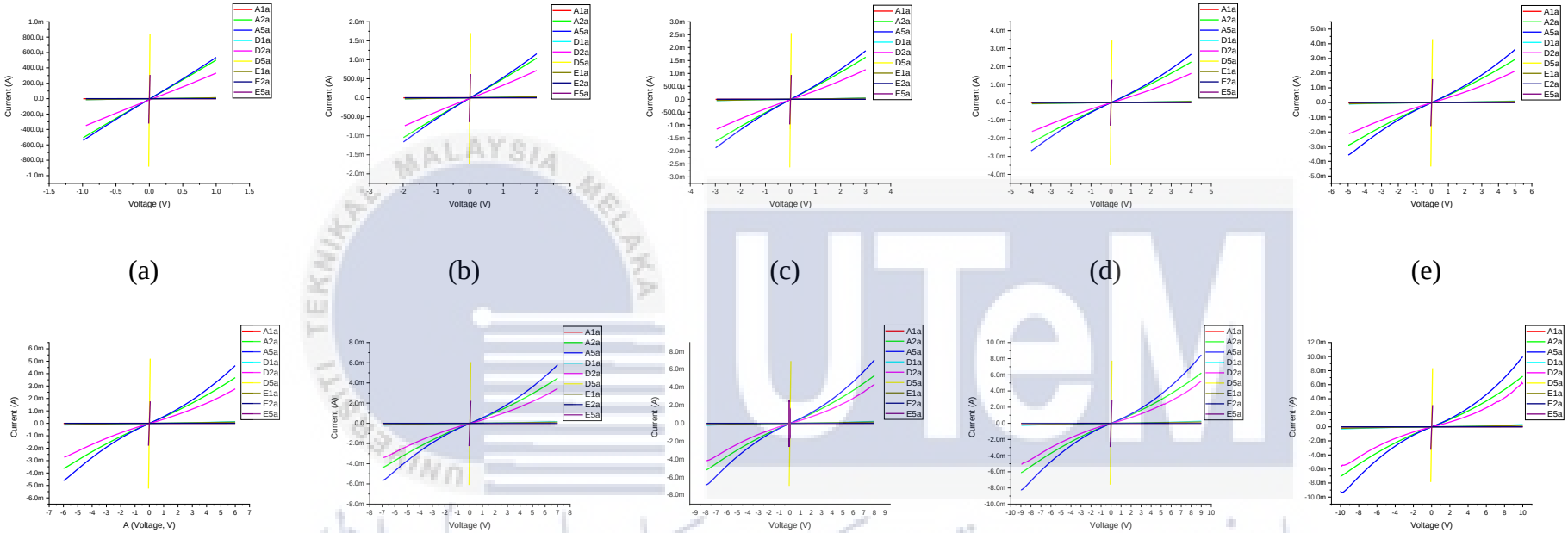
(a) (b) (c) (d) (e)

(f) (g) (h) (i) (j)

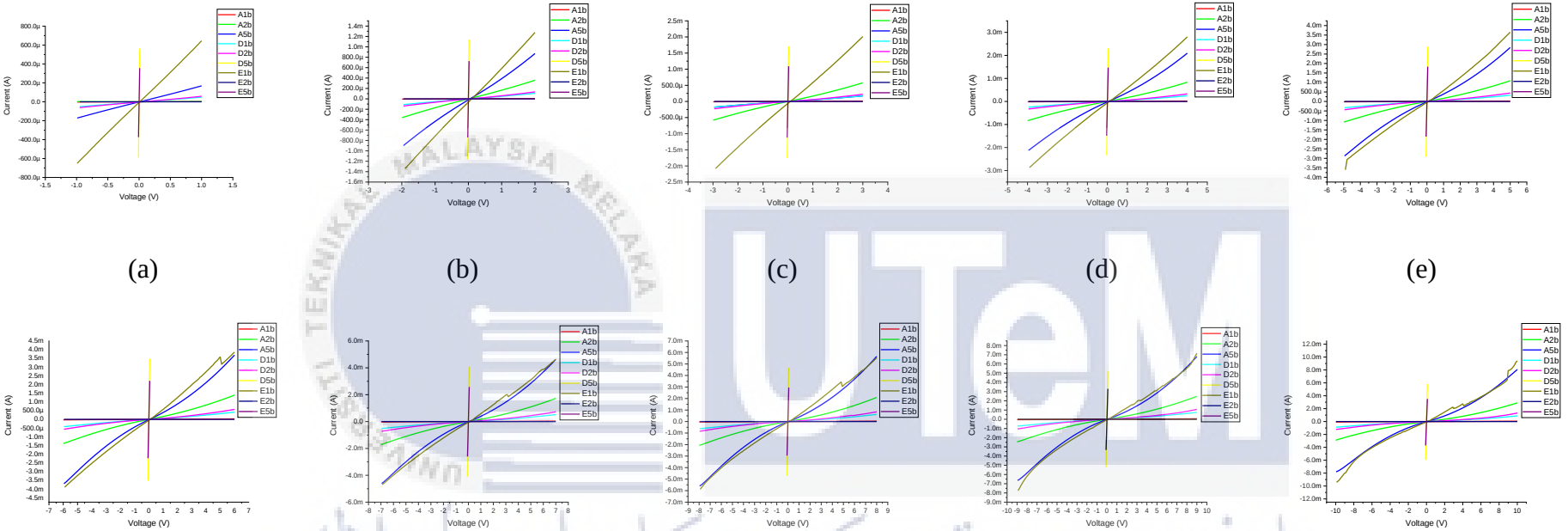
Appendix D: IV graphs with various voltages of gas sensor that using glass as substrate. The gas sensors that will be tested with ethanol gas.



Appendix E: IV graphs with various voltages of gas sensor that using glass as substrate. The gas sensors that will be tested with acetone gas.



Appendix F: IV graphs with various voltages of gas sensor that using Kapton film as substrate. The gas sensors that will be tested with ethanol gas.



Appendix G: IV graphs with various voltages of gas sensor that using Kapton film as substrate. The gas sensors that will be tested with acetone gas.