

NITROGEN DIOXIDE DETECTION USING COPPER OXIDE THIN FILM
SENSOR PREPARED BY DIRECT CURRENT SPUTTERING TECHNIQUE

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*In this thesis, I want to make special dedications to
my lovely family:
Dad, Mohd Sukri Hassan
Mum, Mutia Sobihah Ab Rahim
and all my siblings.*

*Without their love and support, this research would not have been completed in three
years. Here I wrote some poem for them to wish thanks so much for their support;*

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and knowing just what to do.
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when I'm feeling way beyond the blue.*

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to all my worries and stresses.
Thank you for caring enough
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ABSTRACT

Semiconducting copper oxide (CuO) thin films have been deposited using DC sputtering technique using copper (Cu) target for detection of oxidizing nitrogen dioxide (NO₂) gas. A series of CuO thin films (gas sensors) (C1-C7) of various thicknesses had been prepared on Corning glass substrate. The time of deposition was between 90 to 270 minutes at 30-minutes time interval. The thickness of the gas sensors was measured using a surface profiler (Dektak 3) and found to be in the range between 159 nm to 604 nm. The resistance and the gas sensing properties to NO₂, mainly the sensitivity, response and recovery time were investigated using the gas sensor characterization system (GSCS) for a single gas sensor and for an array of sensors in series configurations at different operating temperatures. The resistance of the gas sensor decreased when exposed to NO₂ gas. The best sensitivity (0.859) for the single gas sensor configuration was found to be sensor C3 which was deposited at 150 minutes, corresponding to 247 nm thickness. While, for the sensor array configuration, the best sensitivity (0.788) was found to be sensor AC3 at the combination of 90 and 150 minutes-deposition time. The response time for the gas sensor C3 was 57 seconds and the recovery time was 60 seconds; whereas for the array gas sensor AC3, the response and recovery time were 45 seconds and 60 seconds, respectively. Thus, the single configuration had a better sensitivity and response time compared to the array configuration. Hence, the single configuration was the preferable gas sensor for NO₂ gas.

ABSTRAK

Filem tipis kuprum oksida (CuO) semikonduktor telah dimendapkan menggunakan teknik pemercikan DC menggunakan sasaran kuprum (Cu) bagi mengesan pengoksidaan gas nitrogen dioksida (NO₂). Satu siri filem tipis CuO (penderia gas) pelbagai ketebalan (C1-C7) telah disediakan pada substrat kaca Corning. Masa pemendapan adalah di antara 90-270 minit pada selang masa 30 minit. Ketebalan penderia gas diukur menggunakan pengukuran permukaan (Dektak 3) dan didapati berada dalam julat di antara 159 nm ke 604 nm. Rintangan dan sifat penderiaan gas kepada NO₂, terutamanya kepekaan, masa tindak balas dan pemulihan telah dikaji dengan menggunakan sistem pencirian penderia gas (GSCS) untuk penderia gas tunggal dan untuk pelbagai penderia dalam siri konfigurasi pada suhu operasi yang berbeza. Rintangan penderia gas didapati menurun apabila terdedah kepada gas NO₂. Kepekaan terbaik (0.859) untuk konfigurasi penderia gas tunggal dianalisis pada penderia C3 yang dimendap pada minit ke-150 yang sejajar kepada ketebalan 247 nm. Sementara, bagi konfigurasi pelbagai penderia, kepekaan yang terbaik (0.788) ditemui pada penderia AC3 gabungan minit ke-90 dan 150-masa pemendapan. Masa tindak balas untuk penderia gas C3 adalah 57 saat dan masa pemulihan ialah 60 saat; manakala untuk pelbagai penderia gas AC3, masa tindak balas dan pemulihan masing-masing ialah 45 saat dan 60 saat. Konfigurasi tunggal mempunyai kepekaan dan masa tindak balas yang lebih baik berbanding dengan konfigurasi pelbagai. Oleh itu, konfigurasi tunggal adalah penderia gas lebih baik bagi gas NO₂.

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LIST OF SYMBOLS

Cu	-	Copper
CuO	-	Copper oxide
DC	-	Direct Current
RF	-	Radio Frequency
GSCS	-	Gas sensor characterization system
nm	-	nanometer
°C	-	Degree celcius
Ω	-	Ohm
eV	-	Electron volt
NO ₂	-	Nitrogen oxide
Si	-	Silicon
T	-	Temperature
m	-	Meter
Min	-	Minute
R_{air} / R_a	-	Resistance in synthetic air
R_{gas} / R_g	-	Resistance in NO ₂ gas
V	-	Voltan
V _c	-	Fixed DC voltage
V _L	-	Load voltage
V _{La}	-	Load voltage in synthetic air
V _{Lg}	-	Load voltage in NO ₂ gas
R _s	-	Resistance of the sensor
R _L	-	Load resistance
TiO ₂	-	Titanium oxide
LGS	-	Langasite

SnO ₂	-	Tin oxide
Ppb	-	parts per billion
Ppm	-	parts per million
Au	-	Aurum
RPP	-	Rapid photothermal processing
P	-	Phosphorus
As	-	Arsenic
Sb	-	Antimony
Ge	-	Germanium
B	-	Boron
Ga	-	Gallium
CO	-	Carbon monoxide
H ₂ S	-	Hydrogen sulfide
H ₂	-	Hydrogen
CH ₄	-	Methane
ZnO	-	Zinc oxide
In ₂ O ₃	-	Indium oxide
HCHO	-	Fulminic acid
N ₂ O	-	Nitrous oxide
N ₂ O ₅	-	Dinitrogen pentoxide
O ₂	-	Oxygen
Ar	-	Argon
WO ₃	-	Tungsten trioxide
CVD	-	Chemical vapour deposition
Å	-	Armstrong (1x10 ⁻¹⁰ m)

CHAPTER 1

INTRODUCTION

1.1 Research Background

Semiconductor gas sensors are investigated extensively for the aim of practical applications, like gas leak detection and environmental observance. Several efforts are created within the technology in this field that aims to extend the gas reaction or sensitivity, selection, stability, and therefore the chance for practical use (Noboru et al., 2003). Furthermore, device arrays, even have been used to improve the choice of semiconductor gas sensors. The utilization of an array of sensors combined with pattern recognition analysis is an efficient approach to enhance the choice and form of practical applications (Penza et al., 2001). However, further innovations of gas sensing properties are still in nice demand to expand the fields of gas device applications. (Noboru et al., 2003).

One of the gases that cause the pollution in the environment is nitrogen dioxide (NO₂), known for its pungent odor and irritating and it is often emitted from vehicle

exhaust gas in low concentrations (<500 ppm). As a matter of fact, all fossil fuel combustion in air produces oxides of nitrogen (NO_x), in which NO_2 is the main product. Lasting exposure to low levels of NO_2 was found to increase the risk of respiratory symptoms (Liu et al., 2012). NO_2 was exhausting from automotive engines, boilers, or any other combustion processes of fossil fuels is one of the representative air pollutants, which give rise to environmental problems including photochemical smog and acid rains. For monitoring and controlling NO_2 emission, it is necessary for developing high-performance NO_2 sensor that should continuously detect NO_2 with sensitivity and selectivity (Ling et al, 2013). Sensitivity is the ratio of the resistance in the gas detection, R_g and the resistance in the air, R_a . While selectivity is the characteristic that determine whether a sensor can response selectively to a group in series or in the single sensor.

The NO_2 sensors based on semiconductor metal oxides have widely been investigated. Although these sensors are of high sensitivity, they are usually operated at low temperature (100–400 °C) and their sensitivity will disappear above 500–600 °C. However, in the commercial combustion application, the sensor must withstand high temperatures (600–900 °C) for a long time. Furthermore, poor selectivity caused by cross-sensitivity of NO_x sensors based on semiconductor metal oxides to coexistent gases is a challenge and it consequently limits their utility (Ling et al., 2013).

Nowadays, n-type metal oxides are widely investigated, such as ZnO, In_2O_3 , and SnO_2 , due to their extensive sensing performance. Recently, increasing interest has been taken in gas sensors based on p-type semi-conductors. Among a variety of p-type semi-conductors, copper oxide (CuO) has proved itself to be one of the most attractive materials for gas sensor applications, from the point of view of gas sensitivity as well as chemical stability. CuO has been found to be sensitive to HCHO, NO_2 , H_2S and CO (Gopalakrishna et al., 2013). In contrast, CuO a more stable for sensing toward reducing gases for NO_2 detection (Das et al., 2013). Reducing gas is when it steamed on a metal

oxide semiconductor, the gas reacts with the oxygen ions on the semiconductor surface, releasing electrons back to the conduction band. Therefore, when the concentration of electrons on the semiconductor surface increases, the resistance the semiconductor increases because the generated electrons recombine with holes (Takafumi et al, 2013). There have also been reports of successful conductometric NO₂ sensing with CuO at lower operational temperatures in the order of 200 – 350°C (Micheal et al., 2012).

The CuO is a p-type semiconductor with a narrow band gap of 1.2 eV. Such semiconducting properties make it a potential candidate for solar cell fabrications, catalytic applications and also in gas sensors. Low operating temperature, a cost effective option, offers stability to the active sensing materials and becomes an automatic choice for sensing application. However, for metal oxides, including surface reaction by an adsorption of toxic gas, the recovery process of the sensing materials depends strongly on the operating temperature. Therefore the demand for low operating temperature sensor faces strong challenge that needs further developments, in particular, synthesis and process control of suitable materials. With the advent of synthesis of metal oxides which provide manipulation of the operating temperature and obtaining a superior response, a basic requirement for sensing, becomes feasible (Das et al., 2013).

In this study, copper oxide was chosen to detect nitrogen dioxide, NO₂. Copper oxide is a semiconductor and has been studied for a number of reasons such as natural abundance of starting material (Cu) and the easiness of production by the oxidation of Cu. It also essentially non-toxic and electrical properties and optical properties are quite good (Papadimitropoulos, 2005). Copper oxide (CuO) has unique characteristics such as low cost, non-toxic, abundant availability of copper and the formation of the oxide layer is quite simple (Dhanasekaran et al., 2012). Copper oxide thin films were prepared using a DC sputtering method for gas sensor applications in this study.

1.2 Statement of Problem

Atmosphere of chemicals, particulate or biological materials that cause discomfort, illness, or even death to humans and other living organisms. Air pollution also can cause damage to crops the environment or built environment. Earth always consist of a complex dynamic system of natural gas in the material environment to support all living things on earth. Effects of air pollution and stratospheric ozone depletion has been recognized and a threat to human health and the ecosystem of the earth.

Therefore, controlling and monitoring NO_2 gas is an important task for environmental protection. In this study, there are two problems to be solved related to the environment. The problems are;

- 1) The effect of NO_2 gases on the environment. NO_2 is the toxicity of our health and environment. It needs to control and monitoring its effect in our lives.
- 2) The optimization of thin film technology in order to overcome the problem of poor sensitivity, selectivity and response time and recovery time detection of NO_2 gas.

1.3 Aims of Study

The objectives of this study are as follows:

- 1) To fabricate copper oxide thin film gas sensor samples for nitrogen dioxide detection using DC sputtering technique.
- 2) To determine the sensitivity, response time and recovery time of samples at different operating temperature for single sensor and sensor array configurations for NO₂ detection.

1.4 Scope of Study

In this study, copper oxide thin film were prepared by using DC sputtering technique. Seven samples were prepared at different time of deposition so that every sample had a different thickness. The thickness was measured by using the surface profiler (Dektak 3).

The sensor samples would than be characterize its NO₂ sensitivity. The measurement will be counted and by the use of gas sensor characterization system developed by the previous researcher (Wan, 2006). In this measurement we would monitor the dependence of sensor resistance to the NO₂ concentration. The efficiency of the sensor also would be determine by observing the effect of operating temperature as the NO₂ detection sensitivity.

1.5 Significant of Study

There are many gaseous nitrogen-oxygen compounds, denoted by NO_x , that exist in various states of oxidation, from N_2O to nitrogen dioxide (NO_2) and dinitrogen pentoxide (N_2O_5). When the NO_2 react with oxygen and hydrocarbon, it forms very reactive organic radicals that are strong oxidizing agents and can lead to eye irritation. NO_2 was absorbed sunlight and giving it the brownish colour characteristic of smog.

Therefore, controlling and monitoring NO_2 gas is an important task for environmental protection. The copper oxide thin film was selected to detect toxic gases. Copper oxide is non-toxic and the advantage of using this thin film as the application devices and substances that can be found in the abundance of p-type semiconductor which has the most stable defects in both of the environments Cu and O-rich as their copper vacancy (Ooi et al., 2013).

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