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Correlation between Microstructure of Copper Oxide Thin Films and Its Gas Sensing Performance at Room Temperature

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Abstract

Radio-frequency magnetron sputtering using a Cu target was used to deposit cuprous oxide and cupric oxide thin films on silicon wafer. The substrate bias voltage and the O_2 flow ratio were varied during the deposition. The deposited thin films were characterized using scanning electron microscope. We found that the spherical and pyramid shapes structure of copper oxide thin films were deposited at critical O_2 flow ratio between 7 and 14%. The influence of substrate bias voltage was small and negligible. The deposited thin films were used for sensing characterization using ethanol vapor. Experimental results reveal that the pyramid shape of copper oxide thin film contribute to high respond rate when exposed to ethanol vapor. The respond and recovery rates which were measured at room temperature were very fast. This work had successfully demonstrated the formation of optimized copper oxide thin films and their usage for gas sensing application.

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Keywords: Magnetron sputtering plasma; Thin film; Copper oxide;Gas sensor

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1. Introduction

Copper is one of the material that have being explored for various applications. One of the most promising applications is on the gas sensing characteristic of copper oxide. The advantage of copper is low cost, sensitive to ambient condition and easiness to produce oxide thin film [1–3]. Copper is well known to form two type of metal oxides which is cuprous oxide (Cu_2O) and cupric oxide (CuO)[4–6]. Cupric oxide is a p-type semiconductor that has a narrow band-gap of 1.2eV and low resistance value[4]. Some of the gases that have being reported to be sensed by cupric oxide thin films are NO_2, CO, CO_2, H_2S, ethanol and methanol[7-9].

There are several techniques which are suitable to deposit the copper oxide thin films such as spin coating, electron beam evaporator, pulse laser deposition and reactive magnetron sputtering deposition [1-6]. In this experiment, reactive magnetron sputtering deposition was selected to produce copper oxide thin film due to its purity, simplicity, controllability and repeatability performance. Sputter deposition at specific substrate bias voltage is a technique widely used to control the deposition rate, structural, electrical and optical properties of the films. Since that substrate bias voltage is similar to the heating of substrate, it is expected that the grain size will be different depending on the substrate bias voltage. In addition, the electrical resistivity of films improved due to the enhancement of the crystallity of the thin films and the increase of optical band gap [10]. In order to produce a high sensitivity copper oxide thin film, details investigation on the reactive sputtering deposition is necessary. In the present work, copper oxide thin films were deposited at various substrate bias voltages and O_2 flow ratios. The microstructure and gas sensing performance of copper oxide thin films were evaluated and finally the correlation between the microstructure and gas sensing performance were discussed.

2. Experimental Details

Copper oxide thin films were deposited using magnetron sputtering plasmas at various O_2 flow ratios and substrate bias voltages. The sputtering deposition plasma was produced by 13.6 MHz RF magnetron discharges with an automated matching network. The sputter target was made of 3 inches pure (99.99% purity) copper target. The sputter chamber was evacuated to base pressure less than 10^{-6} Torr using vacuum turbo molecular pump and backed by rotary mechanical pump. The argon and oxygen gases were introduced into the chamber by using mass flow controller that was attached to the top of the chamber. The argon flow rate was fixed at 50 sccm. The oxygen flow ratio ($O_2/(O_2+Ar)$) was controlled by varying the oxygen flow rates from 0 to 16 sccm. In order to produce negative substrate bias voltage into the chamber, a DC power supply was connected to the substrate holder within the chamber and the substrate bias voltages were varied from 0 to -100 V. The total working pressure was fixed at 22.5 mTorr during the whole processes. The RF discharges power was fixed at 400 W. The copper oxide thin film was deposited on SiO_2 coated Si wafer substrate for 4 minutes. The distance between the Cu target and the substrate surface was approximately 13 cm. The microstructure of the deposited films was observed using field emission scanning electron microscope (FE-SEM) operated at 5 kV.

In order to perform an investigation on the response and recovery times of the gas sensor, a simple circuit was used to conduct the measurement. The circuit design and the overview of the gas sensor testing are displayed in Fig. 1. The circuit for measurement is design using the theory of voltage divider rule as follow:

$$V_{out} = \frac{R_P}{R_P + R_{sensor}} V_S \quad (1)$$