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Sputter Deposition of Cuprous and Cupric Oxide Thin Films Monitored by Optical Emission Spectroscopy for Gas Sensing Applications

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Abstract

Cuprous oxide and cupric oxide thin films were deposited on silicon wafer with additional substrate bias voltage using radio-frequency magnetron sputtering of a Cu target with Ar+O_2 discharge plasma. Optical emission spectroscopy was employed to monitor the intensity of atomic Cu and O emission lines at various substrate bias voltages and oxygen flow ratios. Thin film transition from cuprous oxide to cupric oxide phase was observed by X-ray diffraction analysis when the oxygen flow ratio increased. This transition was not influenced by the substrate bias voltage. Optical emission spectroscopy showed a real time monitoring results of the transition from cuprous to cupric oxide thin films. The transition was observed at a critical O_2 flow ratio of 7%. The results proposed a tightly control of reactive Cu sputtering through a closed loop and real-time monitoring system for precise copper oxide thin films deposition.

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1. Introduction

Copper oxide is a p-type metal oxide semiconductor. It is one of the promising candidates to replace the toxic and expensive materials for applications such as dye sensitized solar cell, photo catalysis, photo-chromic devices[1–4]. Recently, nanostructured copper oxide is also considered as a potential field emitter and good gas sensing material[2, 5]. There are two important oxide compound of copper, which is cuprous oxide (Cu_2O) and cupric oxide (CuO). Cupric oxide is a monoclinic semiconductor with indirect band gap range of 1.3-2.1eV[6, 7]. On the other hand, cuprous oxide has a direct band gap slightly above 2 eV. To date, copper oxide thin films have been deposited and growth using several methods, such as radio frequency (RF) magnetron sputtering, chemical vapor deposition, sol-gel and pulsed laser deposition[8–11]. Among those techniques, reactive RF magnetron sputtering is a highly potential method to deposit copper oxide thin films due to its precise controllability and repeatability[12].

Reactive magnetron sputtering of metal targets is a leading industrial process that can uniformly deposit a large area of metal oxide thin films. In general, the characteristic of the deposited copper oxide thin films are influenced by the deposition parameter such as oxygen flow ratio, sputtering power, working pressure and deposition time and substrate bias voltage. It is well known that when oxygen flow supply exceeds a certain critical value, the metal target become poisoned by oxide layer and the sputtering yield changed[13, 14]. In this paper, we developed a controlled deposition process which can grow the multi compound copper oxide thin films with a well controlled properties. It have been reported that the gas sensor response increased when the grain size decreased due to the result from the active adsorption area[16]. Therefore, in order to produce a good sensing device, precise control of grain size of copper oxide thin film is also required.

2. Experimental Setup

Figure 1 show the experimental setup for copper oxide plasma process using RF magnetron sputtering system embedded with the optical emission spectroscopy (OES) system. The plasma chambers consist of six viewports and had a diameter of 16 cm and a height of 18 cm. The magnetron sputtering source was made of cylindrical permanent magnets attached to an indirect water cooling system. The sputtering plasma was produced by a 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 than10^{-6} Torr using vacuum turbo molecular pump and backed by rotary mechanical pump. The sputtering gas, argon and reactive gas, oxygen was 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 total discharge power, chamber pressure and mass flow controller were connected with a personal computer controller unit for remote control the external parameters. The copper oxide thin film was deposited on Si wafer substrate for 4 minutes. The crystal structure of the films was evaluated by X-ray diffraction with Cu Kα radiation.

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