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Influence of oxygen flow rate on the characteristics of the Tungsten Oxide using RF Magnetron Sputtering

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Abstract—Tungsten Oxide (WO3) thin films were deposited using 99.9% pure tungsten target onto ITO substrate using RF magnetron sputtering in the range oxygen flow rates of 30-50%. The influence of the oxygen flow rate on characteristic of WO3 thin films has been investigated. The transmittance, resistivity, crystallite, roughness, and surface morphology were measured by UV-Vis, 2-point probe, X-Ray Diffraction (XRD), Atomic Force Microscopy (AFM), and Field Emission Scanning Electron Microscopy (FE-SEM) respectively. Experimental result showed that the deposition rate of WO3 thin films decreased by increasing oxygen flow rate. A poor crystalinity or more too amorphous of WO3 thin films produces by using various oxygen content. A higher optical transmittance spectrum detected at 30% oxygen content about 86% at wavelength 550nm.

I. INTRODUCTION

WO3 have been studied due to their characteristic on optical transmittance since 1980s with the realization WO3 acting as electrochromic materials. The most promising WO3 widely used in smart glass, gas sensor, automotive rear-view mirrors, and sun roofs [1]. WO3 shows a strong ion intercalation behavior which is this ion insertion is combined with a strong change of the oxide and this effect is exploited intensively in electrochromic (EC) device [2]. Several method for prepared a WO3 have been studied by researcher include vacuum evaporation, pulse laser deposition, magnetron sputtering, spray pyrolysis, chemical vapor deposition (CVD), electrodeposition and sol-gel deposition [3]. RF and DC Magnetron Sputtering process widely used due to better adhesion on the substrate and Sputter deposited films have a composition close to source material. Since the electrical properties, optical properties and microstructure of the sample is depend on the sputtering condition, thus it is important to control sputtering parameter to obtain a good WO3 thin film for EC materials. In this study we examine influence of oxygen flow on WO3 thin film using 30-50% oxygen flow by using flow ratio of O2/(Ar +O2).

II. EXPERIMENTAL PROSEDURE

A. Sample preparation

WO3 thin films were prepared by RF magnetron reactive sputtering using 3-in diameter tungsten target with 99.9% purity by changing oxygen content parameter. The substrate used to receive deposited ion is Indium-tin-oxide (ITO) with sheet resistance 20-80Ω/cm². The area of sample was fixed to 2.5cm x 2.5cm, it cleaned using ultrasonic in acetone and ethanol, and dried in flowing nitrogen gas. The target was sputtered for 10min before fabrication process to clean the target surface from any oxide layer. The fabrication process was conducted in argon and oxygen atmosphere with base pressure of 1 x 10^{-6} Torr and working pressure of 5 x 10^{-3} Torr. The RF sputtering power is 100W and duration for fabrication is 40min with distance target to substrate about 14cm.

B. Characterization

The crystal structure WO3 thin films were studied by X-ray diffraction (XRD). Fe-sem and AFM was performed to measure the crystallite and surface morphology of samples. The optical measurement was performed in the wavelength region of 300nm to 800nm with a UV-Vis spectrometer. Analysis for deposition rate was conducted by using surface profiler machine.

III. RESULT AND DISCUSSION

Fig.1 show the deposition rate for WO3 thin films deposited into ITO substrate using various oxygen flow. The deposition rate of films decreased by increasing oxygen flow rate in the sputtering chamber, at 30% oxygen content WO3 film show highest deposition rate at 1.736nm/min. Besides, higher oxygen content causes smaller incident energy of Ar ions on target in magnetron sputtering chamber, thus produce a gradually decrease of deposition rate [4].

Fig.2 illustrates the abilities of current to flow on WO3 thin film based on resistivity of films. The electrical resistivity of oxide films was calculated from resistance using I-V characteristic machine. Resistivity increased by increasing oxygen flow rate during fabrication process. At oxygen flow rate 50%, the resistivity shows higher value 3.02m.Ω.

A. Structure and composition

The XRD pattern of WO3 thin films was shown in Fig.3. A low diffraction peak shown in the XRD pattern, the film pattern consist of broad hump without discernable WO3 diffraction peak and indicating that the film is poor crystalline, except for diffraction peak of ITO substrate. It indicated that the film is in amorphous state or mixed amorphous and nanocrystalline in nature. The amorphous diffraction pattern may cause by amorphous property and internal stress of WO3 films [4].

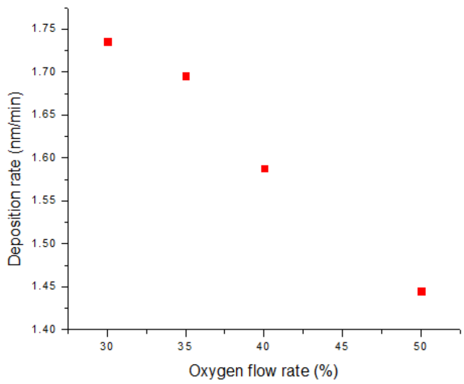

Fig.1. Deposition rate of WO_3 thin films deposited at different oxygen content.

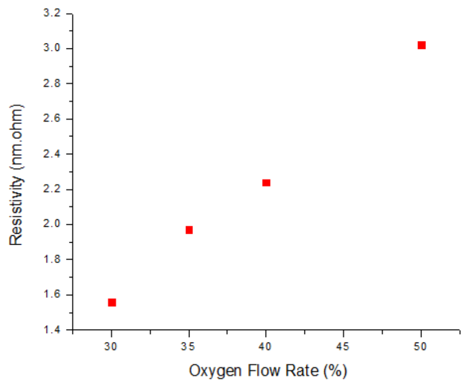

Fig.2. Resistivity change of WO_3 thin films deposited at different oxygen content.

The surface morphological features of WO3 thin films are presented in Fig.4. The WO3 thin film has some big particles distributed randomly on surface, however generally the surface is flat and smooth and this shape clarify that the films is in amorphous state. Besides, Sample fabricated on 45% oxygen flow shows a small distance between particles. These particles consist of formation of tungsten and oxygen which means this sample has small crystalline structure but more too amorphous [5,6].

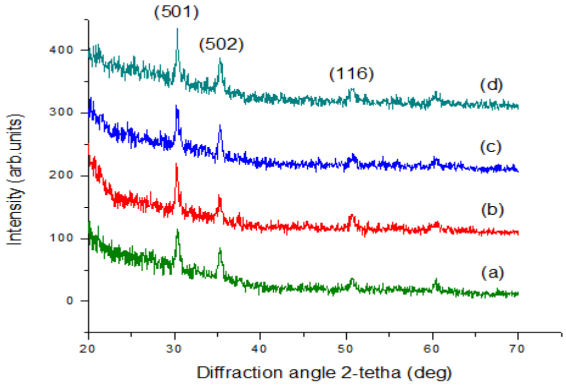

Fig.3. X-ray diffraction pattern of WO_3 films prepared at different oxygen content.

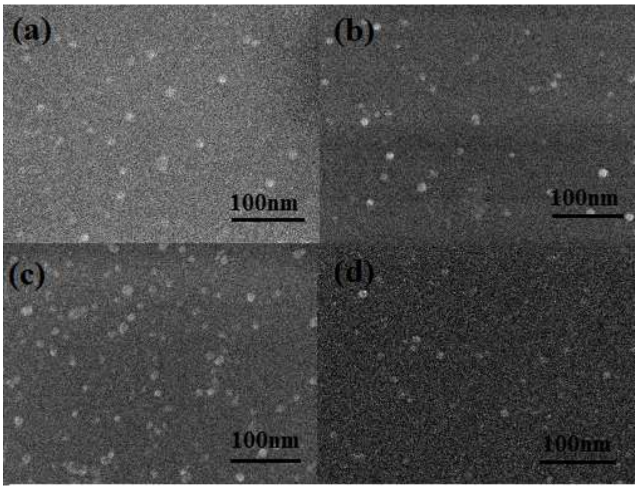

Fig.4. Fe-Sem image of WO_3 film deposited at oxygen flow rate (a)30%, (b)35%, (c)40%, and (d)50.

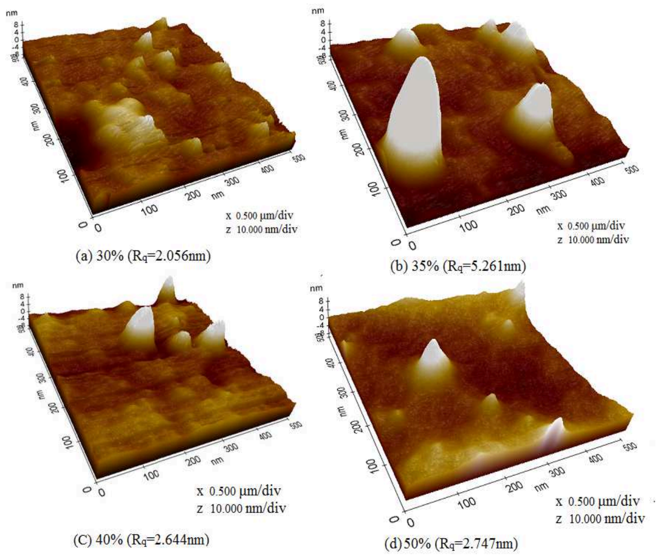

Fig.5. AFM image showing the roughness and surface morphology of WO_3 films deposited at various oxygen flow.

Roughness (δ) of WO3 thin films were measure by using AFM, δ value of thin films from 2.026-5.261nm. Lowest δ is deposited at 30% oxygen and highest δ is deposited at 35% oxygen content. Result indicated that the surface is flat and dense, however miniature hump appeared in several areas. This result shows that variety of roughness is obvious by changing oxygen content in the fabrication process [7].

B. Optical measurements

Fig.6 shows the transmittance spectra of the films at various oxygen flows. The transmittances of 550nm at most film is about 83%, the highest transmittance was detected at 30% oxygen flow about 86% in the visible spectra. From the result, the optical transmittance of WO3 films depends on the oxygen content during fabrication [8].

The transparency of films increased at lower oxygen content, this due to the low stoichiometric process during fabrication. At small oxygen content, the tungsten ion will have a small reaction to oxygen produces a small adhesion onto ITO substrate. Correspondingly, deposition rate of films decreased by increasing oxygen content.

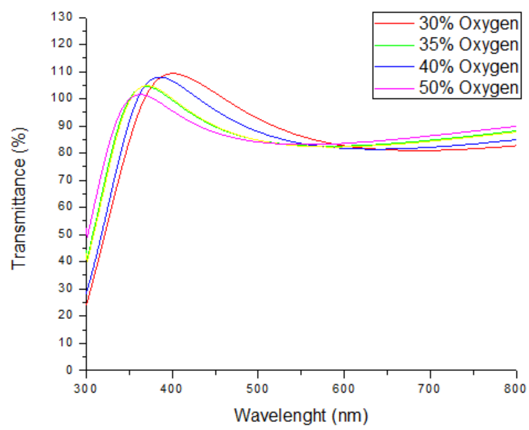

Fig.6. The optical transmittance spectra of the WO_3 thin films deposited on ITO at various oxygen content.

IV. CONCLUSIONS

Amorphous tungsten oxide WO3 thin films were deposited using RF magnetron sputtering by changing oxygen content. The electrical properties, optical properties and microstructure of the films have been studied. The films exhibit better electrochromic properties at 30% oxygen content. However the resistivity and crystallity of films need to be improving for future good. The enhanced electrochromism in these films is attributed to their high thickness about 140nm, small roughness, high crystallity, and have a good optical transmittance.

ACKNOWLEDGMENT

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