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Modulation Of The Transmittance And Conductivity Of Dc Magnetron Sputtered Tungsten Oxide Thin Films By Concentration Variation

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Abstract
Tungsten oxide (WO_3) thin films were deposited using a Direct Current Magnetron sputtering system on glass substrates thoroughly cleaned and mounted on a rotating holder to ensure uniform film deposition. Precursor solutions with WO_3 concentrations of 0.1M, 0.2M, and 0.3M were prepared from sodium tungstate (Na_2WO_4). Deposition was conducted at a base pressure of ~1.0×10^{-6} Torr, working pressure of 3.0×10^{-3} Torr, argon flow rate of 20 sccm, and power of 200 W, followed by a pre-sputtering process and subsequent film deposition at room temperature for 1 hour. Some of the films were annealed at 200 °C for 60 minutes. Optical properties of both the as-deposited and annealed films were measured using a UV-Vis spectrophotometer in the 200-900 nm range. The results indicate that the transmittance was highest in the visible region (350-450 nm) for all films, both as-deposited and annealed, irrespective of concentration. The optical conductivity increased with wavelength from 360 nm to 500 nm and remained constant between 500 nm and 900 nm for all films. Also noticed was that annealing increased the transmittance, which decreased with increasing wavelength, with a more pronounced effect at higher WO_3 concentrations. The increase in transmittance with concentration upon annealing was attributed to film densification, reducing porosity and scattering. Annealing decreased the optical conductivity, likely due to a reduction in free carriers and changes in the material's electronic properties. These findings suggest WO_3 thin films for applications such as smart windows and optoelectronic devices.

***Keywords**: Sputtering, Conductive Oxides, electrochromic, Direct Current, sodium tungstate.*

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I.Introduction

Transparent conductive oxides (TCOs) are materials that exhibit both transparency to visible light and electrical conductivity, essential for optoelectronic devices such as solar cells, touch screens, flat-panel displays, and smart windows (Liu *et al.*, 2010; Hosono & Ueda, 2017; and Afre *et al.*, 2018). Tungsten oxide (WO_3), a pronounced TCO, is valued for its unique electrochromic properties, enabling color change upon the application of an electric voltage, which is ideal for dynamic light modulation in smart windows. WO_3 is a crucial electrochromic material due to its reversibility, coloration efficiency, and cost-effectiveness (Morankar *et al.*, 2024). It is an n-type semiconductor with a bandgap ranging from 2.6eV to 3.3eV, which is a function of growth conditions and deposition techniques (Rydosz *et al.,* 2020). WO_3's various crystal phases make it suitable for applications in catalysis, photoelectrodes, electrochromic devices, and chemical sensors. Its high coloration efficiency and electrochemical stability make it superior to other electrochromic materials. Changes in its optical properties occur when electrons and ions are injected or extracted under an electric field, modifying the electronic structure and shifting the Fermi level (Huang *et al.*, 2015). Various methods such as electrodeposition, magnetron sputtering, spray pyrolysis, and pulsed laser deposition can be used to deposit tungsten oxide. Among these, DC magnetron sputtering was chosen for its ability to produce films with uniform thickness and excellent adhesion to substrates (Li *et al.,* 2022).

GV *et al.,* 2023 investigated tungsten oxide (WO_3) thin films deposited on fluorine-doped tin oxide (FTO) and Corning glass (CG) substrates using DC magnetron sputtering. The study examined the effects of varying annealing temperatures on the surface morphology, structural, electrochromic (EC), and optical properties using techniques such as SEM, XRD, FTIR, Raman spectroscopy, cyclic voltammetry, and UV spectroscopy. The analysis showed that WO_3 thin films annealed at 27 °C, 100 °C, 200 °C, and 300 °C were crack-free, while cracking occurred at 400 °C and concluded that post-annealing significantly influences coloring efficiency, which is crucial for electrochromic applications. Sun *et al.*, 2010 investigated the correlation between the electrochromic performance and the film density of tungsten oxide thin films deposited by reactive DC-pulsed magnetron sputtering at varying working pressures. They found that the optical modulation and coloration efficiency of the films were strongly influenced by the amount of tungsten oxide present. They also found that films deposited at higher working pressures were rougher and had lower density, resulting in a faster electrochromic response. In 2020, Al-Kuhaili investigated a modified form of electroabsorption, where the electric field was applied at the interface between the sample and a transparent conducting layer. This technique was used to study the optical transitions in tungsten trioxide (WO_3). The resultant spectral features were distinctly localized in energy. The experimental spectra were analyzed using Aspnes' thirdderivative model, and the results were interpreted based on theoretical calculations.

This study uses the DC magnetron sputtering method to deposit WO_3 films from a sodium tungstate (Na_2WO_4) compound on glass substrates, with concentrations of 0.1M, 0.2M, and 0.3M. The sputtering was conducted at a working pressure of 3.0×10^{-3} Torr and a base pressure of ~1.0×10^{-6} Torr, followed by annealing at 200 °C for 1 hour. The modulation of the transmittance and conductivity of thin films by concentration variation were investigated.

II.Materials And Methods

Tungsten oxide thin films were deposited using a Direct Current Magnetron sputtering system. At first, the substrates (glass slides measuring 75 mm×25 mm×1 mm each) were cleaned using ultrasonic cleaning in acetone, ethanol, and deionized water, followed by drying with nitrogen gas. The substrates were mounted on a rotating holder to ensure uniform film deposition. Tungsten oxide (WO_3) precursor solutions with concentrations of 0.1M, 0.2M, and 0.3M, were prepared from sodium tungstate (Na_2WO_4) compound. 29.38g, 58.77 g, and 88.15g each of sodium tungstate (Molar mass ≈ 293.83 g/mol) was weighed and dissolved in 800 ml of water and transferred to a 1-liter volumetric flask which was filled up to the 1-liter mark with water. With Base pressure: ~1.0×10^{-6} Torr, Working pressure: 3.0×10^{-3} Torr, Argon flow rate: 20 sccm and Power: 200 W, The tungsten target was pre-sputtered for 10 minutes to remove surface contaminants. Thereafter the deposition was performed at room temperature for 1 hour. The as- deposited tungsten oxide thin films were further annealed at 200 ºC for 60 minutes to improve crystallinity and phase stability (Nworie *et al*., 2024). The optical properties of the as-deposited and annealed thin films were measured using a UV-Vis spectrophotometer in the wavelength range of 200-900 nm, which were then used to determine the optical and solid state properties.

III.Results And Discussion Optical Characterization

UV-VIS-NIR spectrophotometer; an instrument that directly measures sample's transmission was employed to acquire the transmission spectra for the as-deposited and annealed thin films of WO_3 samples across the ultraviolet (UV), visible, and Near-infrared (NIR) regions spanning from 200 to 900 nm. With the %T (percentage transmittance) data, other optical and solid state parameters were subsequently computed (Nworie *et al.*, 2022). Figures 1 – 6 are the Plots of percentage transmittance and optical conductivity versus wavelength for as-deposited and annealed films at 200ºC.

The optical conductivity of the films shown in figures 4 to 6 increased with the wavelength range of 360 nm to 500 nm and remained nearly constant between 500 nm to 900 nm for both as- deposited and annealed films across all concentrations. This is similar to that obtained by (Mahjabin *et al.* (2022) and N'Djoré *et al.*, (2022). Specifically, the highest optical conductivity was observed for the as-deposited 0.2M WO_3 film at 506 nm, followed by the 0.1M WO_3 film. Annealing was found to decrease the optical conductivity of the films. This effect can be attributed to a reduction in the number of free carriers and alterations in the material's electronic properties as the wavelength increases.

IV.Conclusion

In this study, the optical properties of Direct Current Magnetron sputtered Tungsten oxide thin films were investigated. Annealing improves the transmittance of WO_3 thin films, particularly at higher concentrations, due to film densification and reduced porosity. The observed decrease in optical conductivity after annealing suggests that the process reduces the number of free carriers and alters the electronic properties of the material. These findings suggest WO_3 thin films in applications such as smart windows and optoelectronic devices.

Conflict of Interest

The authors of this work have no conflicts of interest at this time.**Data Availability Statement**

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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