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Thermal Treatment Dependent Refractive Index And Optical Absorption Coefficient Of Dc Magnetron Sputtered Tungsten Oxide Thin Films

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Abstract
This study investigates the thermal treatment-dependent optical properties of tungsten oxide (WO_3) thin films deposited using a Direct Current Magnetron sputtering system with 99.95% tungsten as the target material. Tungsten oxide precursor solutions with concentrations of 0.1M, 0.2M, and 0.3M prepared from sodium tungstate (Na_2WO_4) were deposited at room temperature for 1 hour under specific conditions (Base pressure: ~1.0×10^{-6} Torr, Working pressure: 3.0×10^{-3} Torr, Argon flow rate: 20 sccm, Power: 200 W) on ultrasonically cleaned glass substrates mounted on a rotating holder to ensure uniform film deposition. Some of the films were annealed at 200°C for 60 minutes. The optical properties of the films were measured using a UV-Vis spectrophotometer in the wavelength range of 200-900 nm. The results showed that the optical absorption edge of the WO_3 films increased rapidly between 370 nm and 500 nm, gradually continuing towards the infrared region. Upon annealing, absorbance increased in the sample of 0.1M WO_3, indicating enhanced film density, while it decreased in others, suggesting reduced defect density or phase changes. The refractive index of the annealed films was generally lower than that of the as-deposited films in the 380 nm to 500 nm ranges but higher between 500 nm and 900 nm, likely due to changes in oxygen vacancies or other defects. The findings highlight the significant role of thermal annealing in tuning the optical properties of WO_3 thin films and suggest its suitability for applications in smart windows, and photonic devices such as optical waveguides.

***Keywords**: Heat treatment; sputtering; metal oxide; WO_3 thin films; Optical properties*

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I. Introduction

Metal oxides with replicable properties are used in thin film coatings for modern applications such as antireflective and electrochromic coatings, as well as smart windows (Mazur *et al.*, 2021; Mahjabin *et al.*, 2022). Research has shown tungsten oxide (WO_3) a prime candidate for these applications (Subrahmanyam & Karuppasamy, 2007). Tungsten, a transition metal with an atomic number of 74 and atomic weight of 183.85 g/mol, is highly valued in industry and science due to its strength, high melting temperature, hardness, and corrosion resistance (Can et al., 2021; Kramynin, 2021). Tungsten naturally occurs in minerals such as ferberite, hubnerite, stolzite, scheelite, and wolframite. WO_3 is a semiconductor with a wide band gap a ranging from 2.6eV to 3.3eV (Rydosz *et al.*, 2020), making it suitable for use in chemisorption gas sensors. It is extensively studied as a sensor layer capable of detecting gases such as hydrogen, ammonia, nitrogen dioxide, methanol, sulfur dioxide, and methane due to its high selectivity (Nisha & Madhusoodanan, 2013; Can, *et al.*, 2021). Various methods such as spray pyrolysis, pulsed laser deposition, magnetron sputtering, and electrodeposition 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).

Subrahmanyam & Karuppasamy (2007) studied tungsten oxide (WO_3) thin films deposited on glass, ITO-coated glass, and silicon substrates using pulsed DC magnetron sputtering with pure oxygen as the sputter gas. The films were deposited at various oxygen pressures ranging from 1.5×10^{-2} to 5.2×10^{-2} mbar. Their research investigated the influence of oxygen sputter gas pressure on the structural, optical, and electrochromic properties of WO_3 thin films. Among other findings, A high refractive index of 2.1 (at 550 nm) was achieved for films deposited at 5.2×10^{-2} mbar, with the refractive index decreasing at lower oxygen pressures. They attributed the decrease in band gap and increase in refractive index at 5.2×10^{-2} mbar to film densification due to negative ion effects during sputter deposition. Watjen *et al.* (2013) focused on the radiative and optical properties of thin tungsten oxide films across wavelengths from 1 to 20 μm (wavenumbers from 10,000 to 500 cm^{-1}), considering microstructural variations. Four films, each with a nominal thickness of 70 nm, were deposited on silicon substrates using DC magnetron sputtering. The study examined the effects of pre- and post-deposition treatments. Differences in radiative properties between samples were analyzed and linked to the crystalline phases and density of the films. Mahjabin *et al.* (2022) investigated the Optical properties such as transmittance, absorbance, and reflectance of tungsten oxide (WO_x) thin films synthesized via RF magnetron sputtering by varying the sputtering power from 30 W to 80 W, using Ultraviolet- Visible (UV-Vis) spectroscopy. Strong dependencies of all evaluated parameters on sputtering power were observed, highlighting the potential to tailor film properties for specific applications. The films exhibited transmittance above 80%, an optical band energy gap of approximately 3.7 eV, and a refractive index around 2, indicating suitability for optoelectronic applications, particularly in photovoltaic research.

In the present study, we examined the effects of Thermal Treatment on the Refractive index and Optical absorption coefficient of DC Magnetron sputtered Tungsten Oxide thin films at different concentrations.

II.Materials And Methods

Tungsten oxide thin films were deposited using a Direct Current Magnetron sputtering system employing 99.95% tungsten as the target material. 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 °C 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 thenused to determine the optical and solid state properties.

III. Results And Discussion

The refractive index and absorption coefficient of 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 were acquired from the transmission spectra. Figures 1 – 6 are the Plots of absorbance and refractive index versus wavelength for as-deposited and annealed films at 200°C. Figures 1-3 display the optical absorption curves of the as-deposited and annealed WO_3 films within the 370 nm to 900 nm range.

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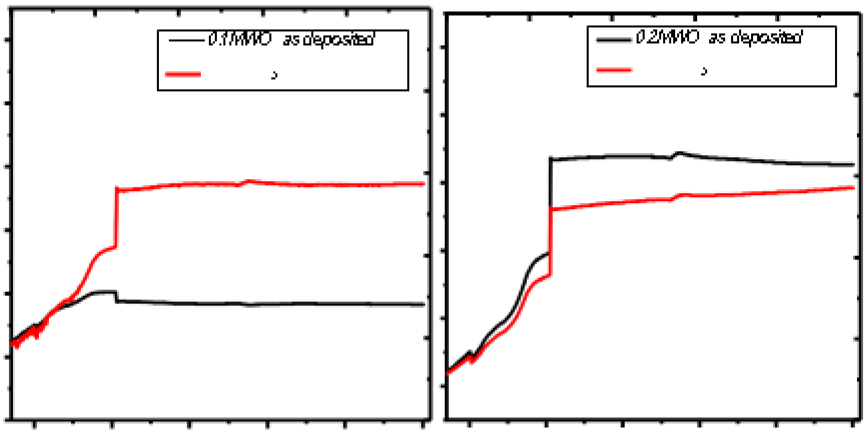

Fig.: 1 Plot of Abs vs Wavelength for as-deposited and annealed 0.1M WO_3
Fig.: 2 Plot of Abs vs Wavelength for as-deposited and annealed 0.2M WO_3

The absorption spectra reveal that the optical absorption edge of the WO_3 films rapidly increases between 370 nm and 500 nm and continues to gradually increase towards the infrared region as the wavelength increases, consistent with the findings of Mahjabin *et al.* (2022). In figure 1, the absorbance value for the as-deposited film is 0.2, which increases to 0.52 upon annealing.

The calculated refractive index characteristics for as-deposited and annealed WO_3 films as a function of wavelength are illustrated in Figures 4 to 6. The analysis reveals that for all concentrations of WO_3 studied, the refractive index of the annealed films is lower than that of the as-deposited films in the 380nm to 500nm range. However, between 500nm and 900nm, the refractive index of the annealed films becomes higher. This could be attributed to alteration by annealing resulting in Changes in oxygen vacancies or other defects which can modify how the film interacts with light across the spectrum. Generally, the refractive index increases with wavelength, consistent with findings by other researchers (Mazur *et al.*, 2021; Mahjabin *et al.*, 2022).

IV.Conclusion

Thermal treatment-dependent optical properties of tungsten oxide (WO_3) thin films deposited using a Direct Current Magnetron sputtering system has been examined. The results indicate a complex interplay between annealing conditions and the optical properties of WO_3 thin films. Thermal annealing plays a significant role in tuning the optical properties of WO_3 thin films. It can both increase and decrease optical absorption depending on specific sample conditions, and it consistently modifies the refractive index across different wavelength ranges. These findings align with previous studies, highlighting the impact of thermal treatment on the structural and optical characteristics of WO_3 thin films. The ability to tune optical absorption and refractive index through annealing makes WO_3 thin films suitable for applications in smart windows, solar cells, photodetectors, and photonic devices such as optical waveguides. Additionally, the controlled refractive index of annealed WO_3 films can be used to design antireflective coatings for lenses and other optical components.

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.