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Synthesis and Characterization of WO_3 thin films deposited by Thermal Evaporation Technique

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Abstract

Thin films of tungsten oxide (WO_3) have been extensively explored as electrochromic materials and have a wide range of uses in electrochromic devices, smart windows, gas sensors, and optical windows. In the present study, the optical, structural and morphological characteristics of WO_3 thin films were investigated. The WO_3 films, were synthesized on glass substrate using the thermal evaporation method under a pressure of 10^{-5} mbar. The prepared films were characterized by X-ray diffractometer, Fourier transform infrared spectroscopy (FTIR), UV–visible photo spectroscopy, Field emission scanning electron microscopy (FESEM).

Keywords: Tungsten oxide, Thermal Evaporation, gas sensors.

Introduction

Recent years have seen a surge in research into metal oxide thin films due to their electrical characteristics, stability at high temperatures, and endurance. Many researchers have investigated metal oxide thin films as electronic materials due to their structural simplicity and low cost. [1] However, to enhance such devices, it is important to completely comprehend the surface flaws and thin film properties that are prevalent during the film fabrication operations. [2] For gas sensing applications, semiconductor metal oxides (WO_3, TiO_2, MgO, and MoO_3) have been extensively Studies. The characteristics and functionality of the gas sensor are anticipated to be improved by these metal oxides with specific nanostructures such nanorods, nanowires, and other nano dimensions [3].

In recent years semiconducting oxide materials like WO_3 have been the subject of intense research due to their low cost, high stability, and ease of sensor fabrication. In recent years semiconducting oxide materials like WO_3 have been the subject of intense research due to their low cost, high stability, and ease of sensor fabrication.[4] Among the transition metal oxides Pure WO_3 and thick coatings of metal tungstate[5] have been found to have exceptional sensitivity to nitrogen oxides at low and high operating temperatures, respectively. [6] Tungsten oxide (WO_3) is an n-type wide band gap metal oxide semiconductor. Due to its intrinsic electrical conductivity, good sensitivity, and selectivity toward various gases like NO_2, WO_3 has emerged as a potential material for gas sensing devices, joining other metal oxides including SnO_2, TiO_2, In_2O_3, and ZnO. [7-8] It is well known that the deposition method and preparation conditions affect the film characteristics. Different techniques, including sol-gel, electro deposition, pulsed laser, sputtering, and spray pyrolysis, were used to prepare WO_3 thin films. [9-14] Additionally, there are conventional methods including thermal evaporation, chemical deposition, and electron beam evaporation. [15-17] In this research, WO_3 thin films were deposited using the high vacuum thermal evaporation from WO_3 powder. The obtained thin films are characterized by using x-ray diffractometer (XRD), UV-Visible spectrophotometer and field emission scanning electron microscopy (FESEM).

Methodology

Synthesis of tungsten oxide thin films:

Tungsten oxide thin films were synthesized by a thermal evaporation technique. The commercially available high purity WO_3 powder (99.9%) was deposited silicon substrates by evaporation in a high vacuum chamber using a molybdenum boat filament. Before the deposition, the powders were placed in desiccator to avoid any moisture and decontamination. Ultrasonically cleaned glass slides were used as substrates. The substrates were mounted on a substrate holder which was placed at a distance of 30 cm in line of sight from the evaporation source. The tungsten trioxide (WO_3) thin films were prepared at room temperature (300K) by the thermal evaporation techniques using a commercial vacuum coating unit (Hind Hivac 12A4D). A diffusion pump backed by a rotary pump was employed to produce the ultimate pressure of 10^{-5} mbar. Deposition was carried out at 10^{-5} mbar. The tungsten oxide got evaporated and directly deposited over the substrate kept at low temperature region. The samples were mounted on a stainless-steel block on a transfer arm. The system was allowed to achieve high vacuum. The molybdenum boat holding the WO_3 powder was slowly heated. The WO_3 powder inside the boat vaporized when the power was applied and the the vapour phase was condensed on the substrate at room temperature. The structural, optical and morphological studies were performed on synthesized thin films.

Characterization Techniques:

The crystalline structure of as-deposited WO_3, films were studied with an X-ray diffractometer. X-ray diffraction patterns of the thin films deposited on glass substrates were recorded by Rikagu miniflex diffractometer equipped with Cu-Kα radiation (λ=1.54056 Å) in a wide range of Bragg's angles 2θ from 10° to 80° at a scanning rate of 2° per minute. The field-emission scanning electron microscopy (FESEM) was carried out to characterize the surface morphologies of the prepared WO_3 thin films deposited on glass substrates. The optical absorption and transmission in the wavelength range 200 – 800 nm were recorded by using a double beam spectrophotometer (UV-VIS Shimadzu). The phase composition was investigated by Fourier Transform Infrared spectroscopy using Vertex 70 spectrophotometer, with a resolution of 4 cm^{-1} over a wavenumber range of 400-4000 cm^{-1}.

Result and Discussion

Structural Properties:

1. X-Ray Diffraction (XRD)

To determine the crystalline phase and crystallinity, the structural characteristics of WO_3 were investigated by using X-ray diffraction (XRD). The XRD pattern of the deposited WO_3 thin films on glass substrate is shown in figure 1. It demonstrates that there are no clear diffraction peaks of the crystalline tungsten oxides, only a widened peak with an amorphous characteristic in the range of Bragg's angles 2θ from 10° to 80° is observed. This is consistent with previously published research. [18-19] The XRD analysis indicates that the as-prepared thin films almost consist of amorphous nature of tungsten trioxide. This may be explained by the fact that for WO_3 thin films crystal lattice alignment and crystallization are not initiated on amorphous glass substrates at ambient temperature. [19].

2. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR measurement was taken to identify the various functional groups that were present in the sample and to learn more about its crystal structure. Figure 2 is the infrared spectrum of WO_3 reveals a broad band with distinctive frequency vibrations between 400 and 4000 cm^{-1}. [20-[21] The band at 540 cm^{-1} is attributed to W–O stretching vibrations, while the band at 626 cm^{-1} is assigned to the W-O-W bridging modes of the WO_6 (octahedral) corner sharing species. The band at 918 cm^{-1} is observed in the range of (Metal=Oxygen) vibrations, which is characteristic for stretching vibrations of W=O bond.

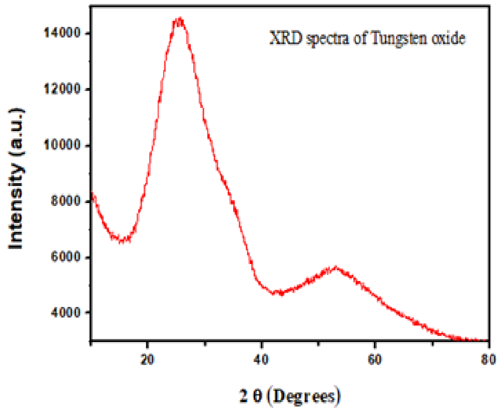

Figure 1: X-ray diffractogram of WO_3 thin films

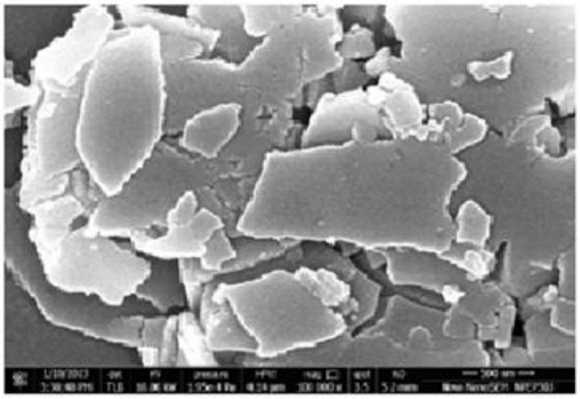

Figure 3. FESEM image of tungsten oxide thin film

Figure 2: FTIR spectrum of WO_3

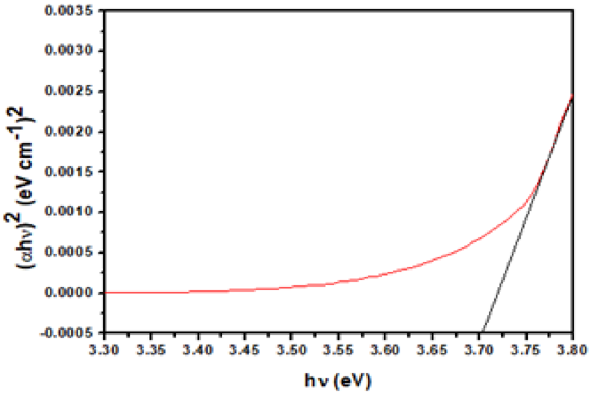

Figure 4. Tauc Plot of tungten oxide thin film

a. Morphological Properties:

The field emission scanning electron microscope has been used to examine the surface morphology of WO_3 thin films (FESEM). Figure 3 shows a FESEM image of the WO_3 thin film. The formation of stacks of WO_3 platelets on the glass surface with various sizes is depicted in FESEM image of deposited WO_3 thin films. [22] These revels the layered growth of the WO3 thin film on the surface. The platelet formation of WO_3 thin film on glass surface was described by Karuppasamy et al. [23].

b. Optical Properties:

In the wavelength range of 200-800 nm, the optical absorption spectra of WO_3 films have been recorded. Figure 4 shows the Tauc plot of $(\alpha hv)^2$ versus photon energy (hv) of the deposited WO_3 thin film, where h is the planks constant, v is the frequency of the incoming radiation, and α is the absorption coefficient. Optical bandgap of the WO_3 thin film was calculated from the linear portion of the Tauc plot. Linear region is clearly seen in figure at $hv > 3.5$ eV for WO_3 thin film which is in accordance with the well-known absorption law $\alpha hv \sim (hv - E_g)^{1/2}$ being characteristic for direct optical transitions. [24] Estimated value of bandgap of the WO_3 thin film is found to be 3.70 eV and is close to the reported value. [25]

Conclusion

In a present study we have carried out deposition of WO_3 thin film by the thermal vacuum evaporation technique on glass substrates. The structural, surface morphological and optical properties of the deposited films were studied. The structural properties of deposited film were investigated by using XRD. The XRD patterns clearly indicates that the prepared WO_3 thin films have amorphous characteristics. FTIR bands present more evidence of the best quality and the high purity of WO_3 thin films. The surface morphological study shows the formation of platelets of WO_3 on the glass surface with different sizes. The UV-Visible characterization revels that optical bandgap of the WO_3 was found to be 3.7 eV at ambient temperature.

This method of thermal evaporation can be used for synthesis of thin films of other metals, alloys and oxides by optimizing parameters like current, temprature, source to substrate distance.

Future research involved the deposition of various metal oxide thin films, including SnO_2, TiO_2, InO2, MoO_3, CeO2, etc., on conducting and non-conducting substrates for a variety of purposes, including gas sensors, solar cells, optical coatings, etc.

Conflicts of interest: The authors stated that no conflicts of interest.

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