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Structural, Morphological and Optical Properties of Electrodeposited Vanadium Oxide Thin Film

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ABSTRACT

Vanadium oxide thin film was synthesized by a simple electrodeposition method. Structural and morphological analyses revealed that the deposited vanadium oxide is polycrystalline in nature with flower like nanostructure. Elemental analysis of the synthesized vanadium oxide thin film showed the presence of desired elements in the required stoichiometry. The optical study of the vanadium oxide thin film was carried by means of reflectance spectra. The UV-Visible reflectance study showed that the reflectance of VO thin film is in the range of 15 to 45 %.

Keywords : Electrodeposition, Vanadium Oxide, Thin Films, Optical Properties

I. INTRODUCTION

Vanadium oxide based materials have some special requirement because of specialty as Vanadium based layered oxides with tunable oxidation states (V^{5+}, V^{4+} and V^{3+}) such as VO_2, V_6O_{13} and V_2O_5 and hence they acts as potential electrode materials for electrochemical devices. Among these oxides vanadium pentoxide (V_2O_5) is better materials than other vanadium oxides because of their high theoretical capacities and fast redox reactions. V_2O_5 generally synthesized in bulk as well as thin film forms. In perspective of electrochemical reactions at electrode-electrolyte interface bulk V_2O_5 has some drawbacks as slow diffusion rate of electrolytic ions, structural instability and low electronic conductivity [1]. In order to improve the performance of V_2O_5 in electrochemical reactions, there has been an increased interest in developing microstructures and nanostructures of V_2O_5 through various chemical methods.

Vanadium oxides have been prepared by various physical and chemical methods such as hydrothermal growth [2], sputtering [3] chemical vapor deposition [4], pulsed laser deposition [5], sol-gel [6], atomic layer deposition [7] and electrodeposition [8]. Among these, electrochemical synthesis has many specialty over the other methods such as its very simple, not expensive equipments are required, less quantity of precursor solution is required, at room temperature or even below and above room temperature synthesis is possible, no toxic chemicals are required, deposition parameters controls the quality of film etc. In this paper we present an inexpensive and easy-to-process

electrodeposition method to produce vanadium oxide thin film.

II. EXPERIMENTAL DETAILS

2.1 Film deposition:

Table 1: Optimized parameters 548

Details	Optimized values
Deposition bath (medium)	Aqueous
Bath composition	0.05 M $VOSO_4.H_2O$ + 2 to 3 drops of concentrated HNO_3
pH	~ 2
Deposition Potential	1.5 V
Deposition time	2 min
Temperature	343 K
Substrate	Stainless steel

We have prepared a deposition bath consisting of vanadyl sulphate for the electrodeposition of vanadium oxide thin film electrodes on stainless steel substrate. The pH of the bath was adjusted by adding 2 to 3 drops of concentrated HNO_3. Preparative parameters, like precursor concentration, deposition potential and deposition time were being optimized to obtain good quality of vanadium oxide thin film. The details of optimized parameters are shown in Table 1.

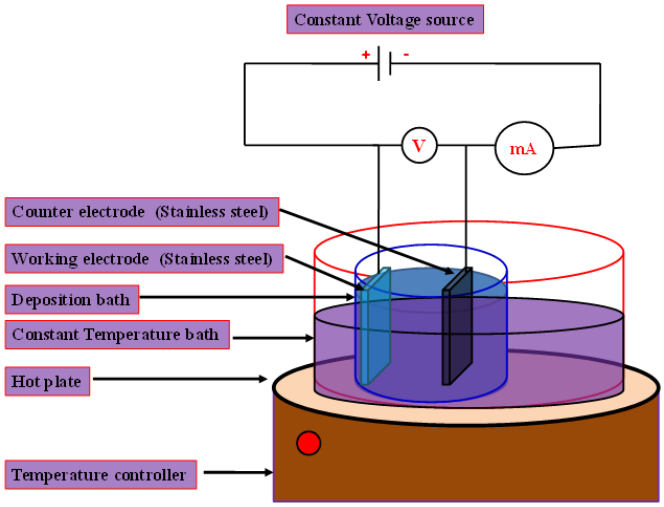

Figure 1: Schematic representation of experimental setup for electrodeposition of vanadium oxide thin film.

We have used two electrode systems for electrodeposition of vanadium oxide thin film. In two electrode system of electrodeposition, working electrode was stainless steel (SS304) and counter electrode was graphite. The deposition bath was maintained at constant temperature of 343 K. Figure 1 represents the schematic representation of actual experimental setup for electrodeposition of vanadium oxide thin film. All other rest of the deposition parameters is mentioned in Table 1 and they are kept constant during the experiment. After deposition films were rinsed with distilled water to remove excessive growth of the film ad kept for drying in air. The dried film is used for characterizations.

2.2 Characterization techniques:

Crystal structure of the electrodeposited vanadium oxide thin film was studied by using XRD in the range of diffraction angle 2θ from 20–80^0 by using Rigaku D/max 2550V 18 kw with Cuk$_\alpha$ diffractometer. The SEM images and EDX analysis were used to study the surface morphology and elemental analysis of the vanadium oxide thin film, respectively. UV-Visible reflectance study of vanadium oxide thin film was studied by UV-Visible spectrometer.

III. RESULT AND DISCUSSION

3.1 X-Ray diffraction study:

X-ray diffraction techniques were used to investigate the crystalline structure of vanadium oxide (VO) thin film deposited on stainless steel substrates by electrodeposition method. Figure 2 represents the X-ray diffraction (XRD) pattern of VO thin film. We can observe that the deposited film has a polycrystalline structure prepared by the electrodeposition method. VO thin film shows four diffraction peaks at 2θ values of (34°, 44°, 50° and 65°). These were assigned to the diffraction lines produced by (022), (114), (062) and (241) of the crystal planes of V_2O_5 confirmed by standard data. All the diffraction peaks in the pattern corresponding to the

orthorhombic crystal structure of V_2O_5 (JCPDS No. 41-1426). From the XRD pattern and comparing with standard data, we have the orthorhombic crystalline phase (space group: Pmmn 59) of V_2O_5 with a lattice constants values of a=11.5 Å, b=3.5 Å, and c=4.3 Å [9]. The characteristic peaks of XRD patterns are well suited with the crystalline V_2O_5 [10]. The average crystallite size was calculated using equation (1), as Scherrer formula [11].

$$D = \frac{0.9\lambda}{\beta.\cos\theta} \quad \text{---- (1)}$$

Where θ: is the Bragg's angle of the XRD Peak. β: Full Width at Half Maximum (FWHM) measured in radians. It can be seen that the estimated crystallite size was found to be 7.8 nm. In literature, also we found the amorphous nature of vanadium oxide thin film. Wu and Lian [12] have reported electroless deposition of vanadium oxide thin film for supercapacitor application. They have been reported that deposited oxide is a mixture of amorphous V_2O_5 and VO_2.

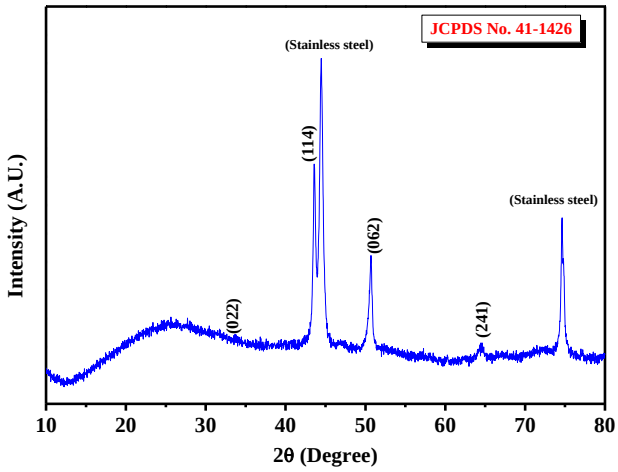

Figure 2: XRD pattern of electrodeposited VO thin film

3.2 Surface morphological study:

The morphologies of VO thin films were analyzed using a SEM. SEM images of the coatings grown using potentiostatic deposition are shown in Figure 3. It mainly consists of some flakes like structures with elongated structures of irregular size and shape. This irregular morphology points to a more textured surface of sample deposited for 2 minute. From the images it is observed that most of the substrate surface is not covered with depositing materials. But it is expected that 2 minute deposition time is not sufficient to coat the whole surface of substrate. Our intension is to study the morphological structure of the film during the formation of vanadium oxide film (at initial stages i.e. formation of coalescence). This study will give the idea of formation of complete microstructure or nano structure. Sui and et al [13] have reported that as deposition time increases, Cu deposition mass increased gradually, and the deposition structure changes from cauliflower-shaped to fine pine branches, finally into coarse pine branches. So, it's interesting to understand the formation of solid phase at very low deposition time.

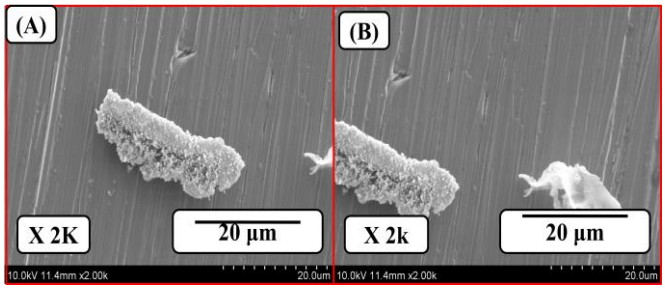

Figure 3: SEM images of electrodeposited VO thin film

3.3 Elemental analysis study.

The elemental composition of electrodeposited vanadium oxide film was analyzed by EDX that confirmed the presence of desired elements in the required stoichiometry as shown in Table 2. This reveals that formation of vanadium pentoxide thin films.The lack of appearance of any peaks other than V and O is a good indicator that the V_2O_5 is homogeneous in composition and formed from V–O. Also, EDX analyses from different regions gave the same outcomes. For the vanadium oxide thin film, the quantitative atomic ratios of V/O is close to expected stoichiometry. Nazemiyan and Jalili [14] have reported that record low temperature Mo doped V_2O_5 thermochromic thin films for optoelectronic

applications. They have reported that elemental composition of electrodeposited films. Our results well match with this literature.

Table 2 : Elemental analysis of electrodeposited VO thin film

Element	Weight%	Atomic%
O K	47.16	73.97
V K	52.84	26.03

3.4 Reflectance spectra study.

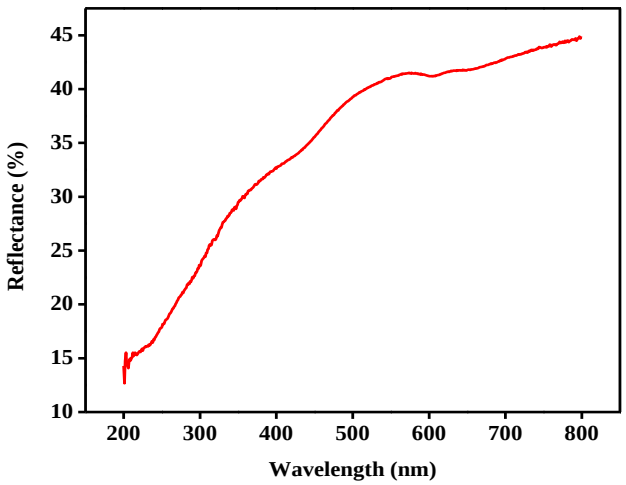

Figure 4 : UV-Visible reflectance spectra of electrodeposited VO thin film.

The reflectance spectra for VO thin film is shown in Figure 4. From the Figure 4, it is observed that the reflectance of VO thin film is > 15 % in the visible and near-IR region. It can be seen that with increasing wavelength, there are obvious increase in the value of reflectance. It is well known that the reflectance is closely related to the properties of surface atoms and density of materials. The reflectance spectra for VO thin film is obtained by equation (2) [15].

$$R(\lambda) = \left(\frac{I_{fr}}{I_m}\right).R_m \qquad \text{--- (2)}$$

Where I_{fr}, I_m are the intensities of light reflected from the sample and the reference mirror, respectively, and R_m is the mirror reflectance. Also, the reflectance depends upon the packing density of unit cell. From Figure 3, it is observed that the reflectance of VO thin film is in the range of 15 to 45 %. This is may be due to formation of closely packed unit cell of VO thin film, which is confirmed by XRD study.

IV. CONCLUSION

In summary, we have successfully deposited vanadium oxide thin film by potenetiostatic mode of electrodeposition. XRD study confirmed the polycrystalline nature of electrodeposited vanadium oxide thin films. SEM images showed that the formation of coalescence on the substrate i.e. formation of solid from liquid phase. EDX analysis confirmed the presence of desired elements in the required stoichiometry. From reflectance study it is observed that the reflectance of VO thin film is in the range of 15 to 45 %.

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