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Effect of Deposition Time on Structural, Morphological and Surface Reflectance Properties of Electrodeposited Vanadium Oxide Thin Films

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ABSTRACT

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Vanadium oxide thin films were prepared by potentiostatic mode of electrodeposition method. The effect of deposition time on the structural, morphological and surface reflectance properties was studied by means of X-ray diffraction study, Scanning electron microscopy and UV-Visible reflectance spectroscopy. Structural and morphological analyses revealed that the deposited vanadium oxide is polycrystalline in nature with porous nanostructure. The surface reflectance study of the vanadium oxide thin films was carried by means of reflectance spectra. The UV-Visible reflectance study showed that the reflectance of VO thin film is in the range between 5 to 45 %.

Keywords : Electrodeposition, X-ray diffraction study, Vanadium oxide, thin films, reflectance spectra

I. INTRODUCTION

In the field of science and technology, transition metal oxides have been broadly studied thanks to their wide variety of electrical, optical and magnetic properties, which made them capable materials for a variety of applications. Amongst of all transition metal oxides, vanadium oxides has taken a prominent position i.e. recently many researchers have been focused on vanadium oxides because it has some special properties and hence used in various fields.

In the literature few vanadium oxides has been reported. But V_2O_5 has been extensively studied because V_2O_5 has layer structure, excellent thermoelectric property and electrochemical activity, good specific energy, high theoretical specific capacity, multiple electron transfer ability, rich resource and low cost and capability of storing intercalating ions. V_2O_5 has some polymorphs, such as α-V_2O_5 (orthorhombic), β-V_2O_5 (monoclinic or tetragonal) and γ-V_2O_5 (orthorhombic). Such properties of V_2O_5 play an important role in applications. Hence V_2O_5 based samples used in various applications such as electrochromic devices

[1], sensors [2], photo-catalyses [3], lithium ion battery [4-5], actuators [6], supercapacitors [7] etc. Numerous physical and chemical methods have been adopted for the synthesis of V_2O_5 like hydrothermal growth [8], Laser induced plasma method [9], chemical vapour deposition [10], sol–gel [11], reactive sputtering method [12], vapor transport process [13] and electrochemical deposition [14]. Among these methods, electrodeposition process has some benefits than other synthesis techniques as cost effectiveness, low operational temperature, simplicity, less quantity of precursor solution is required, negligible waste material and no need of additives, deposition parameters controls the quality of film etc.

Yunus and et al [15] have reported structural studies of ZnO nanostructures by varying the deposition parameters. They have been reported that as deposition time increases the films thickness, length of nanorods and diameter of nanorods increases. Lalasari an et al [16] have reported electrical, optical and structural properties of FTO thin films fabricated by spray ultrasonic nebulizer technique from $SnCl_4$ precursor. In this work they have reported that as deposition time increases, peak intensity in XRD pattern increases and electrical conductivity decreases. In this paper we present an inexpensive and easy-to-process electrodeposition method to produce vanadium oxide thin film. An effort has been taken to study effect of deposition time on structural, morphological and surface reflectance properties of electrodeposited Vanadium Oxide thin films.

II. EXPERIMENTAL DETAILS

2.1 Film deposition:

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. 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 temperature were being optimized to obtain good quality of vanadium oxide thin film. All other rest of the deposition parameters are kept constant during the experiment, mentioned in Table1. 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.

Table 1: Optimized parameters

Details	**Optimized values**
Mode	Potentiostatic
Deposition Potential	1.5 V
Bath composition	0.05 M $VOSO_4.H_2O$ + 2 to 3 drops of concentrated HNO_3
pH	~ 2
Deposition bath (medium)	Aqueous
Deposition time	(a) 120 s; (b) 240 s; (c) 360 s.
Temperature	343 K
Substrate	Stainless steel

2.2 Characterization techniques:

Crystal structure of the electrodeposited vanadium oxide thin films was studied by using XRD in the range of diffraction angle 2θ from 20–80^0 by using Rigaku D/max 2550Vb+ 18 kw with Cuk_α diffractometer. The SEM images were used to study the surface morphology of the vanadium oxide thin film. 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:

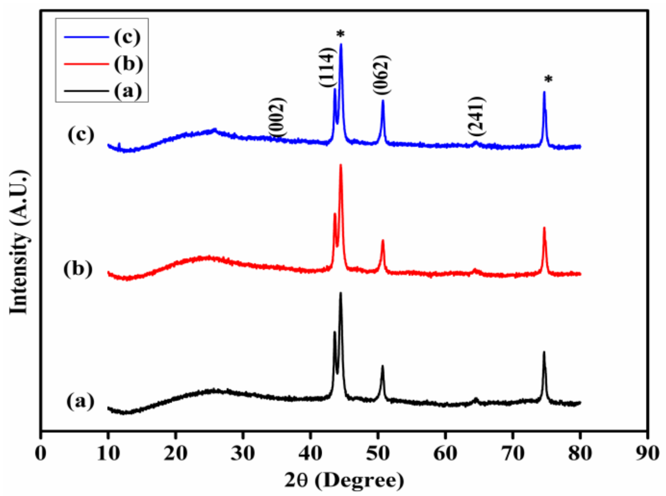

Figure 1: XRD pattern of electrodeposited vanadium oxide thin films for film deposited at a) 2 min; b) 4 min and c) 6 min.

Figure 1 illustrates XRD patterns of vanadium oxide thin films deposited at various deposition times ranging from 120 to 360 s. All the patterns exhibited three broad peaks, which appear at 2θ values of 34°, 44°, 50° and 65°. According to the standard diffraction data (JCPDS: 41-1426), these diffraction peaks correspond respectively to the (022), (114), (062) and (241) planes of an orthorhombic structure of vanadium oxide. From Figure 1, it is observed that the crystallinity of the flm increases with increase in deposition time. The intensity of the (062) plane is significantly higher, indicating that the crystallites have grown more in this direction and the films of vanadium oxide preferentially occur along the (062) plane. At deposition time 360 s, the broadening of (062) peak revealed the formation of nanometric nature of vanadium oxide. The samples deposited from 120 to 360 s do not show any additional peaks, which indicates no secondary phase was formed. The average crystallite size of the vanadium oxide thin film was estimated from the full width at half maximum (FWHM) according to the (062) plane using Debye- Scherrer equation (1) [17]:

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

Where, λ is the X-ray wavelength, β is the full width at half maximum of the XRD peak and θ is the Bragg diffraction angle. These results point out that by increasing deposition time from 120 to 360 s, the size of the crystallites increases from 7.8 nm to 9.4 nm.

3.2 Surface morphological study:

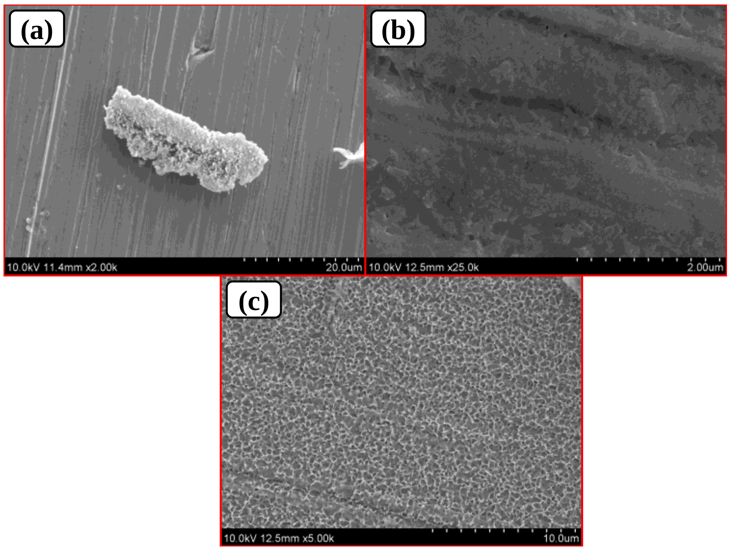

Figure 2: SEM images of electrodeposited vanadium oxide thin films for film deposited at a) 2 min; b) 4 min and c) 6 min.

The surface morphologies of the vanadium oxide thin films obtained with different deposition times at 343 K were determined by using scanning electron microscopy (SEM) and are shown in Fig. 2. When the SEM patterns of the films were examined, it was observed that as electrodeposition time increased from 120 s to 360 s, the number of coalescence in the film increased and more surface of substrate covered; while, it was observed that because of the clusters collections (ion-by-ion mechanism) and clusters growth (cluster-by-cluster mechanism) on the surface of substrate a porous structure formed. These results are in good agreement with the literature. Haddad and et al [18] have reported that effects of deposition time in chemically deposited ZnS films in acidic solution. They reported that as deposition time increases film structure becomes dense and uniform that was free of pits or pinholes.

3.3 Reflectance spectra study.

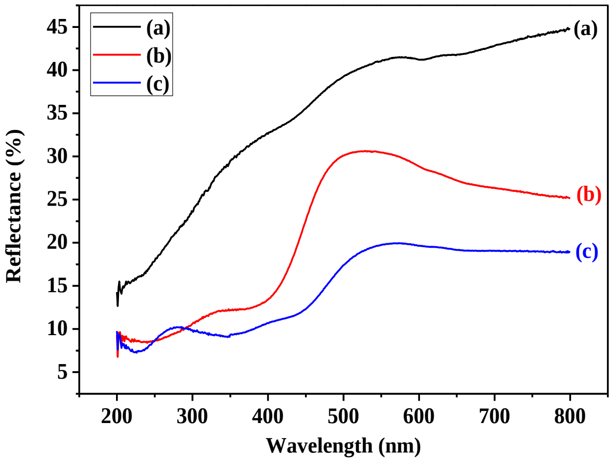

Figure 3: Reflectance study of electrodeposited vanadium oxide thin films for film deposited at a) 2 min; b) 4 min and c) 6 min.

The reflectance spectra for VO thin film is shown in Figure 3. From the Figure 3, it is observed that the reflectance of VO thin film is > 5 % in the visible and near-IR region, for deposition time of 120 s to 360 s. It can be seen that with increasing wavelength, there are obvious increase in the value of reflectance for all samples. It is well known that the properties of surface atoms and density of materials will affect the reflectance of the sample. The reflectance spectra for VO thin film is obtained by equation (2) [19].

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

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 5 to 45 %. This is may be due to formation of closely packed unit cell of VO thin film, which is confirmed by XRD study. Also, From SEM study it is observed that there is formation of cluster and cluster aggregations on surface of substrate.

IV. CONCLUSIONS

In summary, we have successfully deposited vanadium oxide thin film by potenetiostatic mode of electrodeposition with deposition times between 120 s to 360 s. XRD study confirmed the polycrystalline nature of films with improved crystallinity. SEM images showed that the clusters collections (ion-by-ion mechanism) and clusters growth (cluster-by-cluster mechanism) on the surface of substrate a porous structure formed. From reflectance study it is observed that the reflectance of VO thin film is in the range of 5 to 45 % due to close packing of unit cell.

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