Study of structural, electrical and optical properties of vanadium oxide condensed films deposited by spray pyrolysis technique

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Abstract Vanadium oxides condensed films are prepared on glass substrates, using spray pyrolysis (SP) technique. The effects of substrate temperature, vanadium concentration in initial solution and the solution spray rate on the nano-structural, electrical, and optical properties of deposited films were investigated. Characterizations of the samples were performed using X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV-Vis spectroscopy methods. The type and concentration of the charge carriers were determined and measured by Hall effect experiment. XRD patterns showed that the prepared films had polycrystalline structure, mostly tetragonal β - V_2O_5 and V_4O_9 phases with the preferred orientation along (200) direction. The Hall effect experiment revealed that all samples were *n*-type, except the ones deposited at substrate temperatures $T_{\text{sub}} = 300^\circ\text{C}$ and $T_{\text{sub}} = 450^\circ\text{C}$, vanadium concentration 0.1 mol/L and solution spray rate 10 mL/min. The charge carrier concentrations obtained were in the range 10^{16} – 10^{18} cm^{-3} . The lowest sheet resistance (R_s) was obtained for the samples prepared at $T_{\text{sub}} = 450^\circ\text{C}$, vanadium concentration 0.05 mol/L and solution spray rate 10 mL/min. It was also found that the optical transparency of the samples changed from 20% to 75% and the optical band

gap of the samples was from 2.22 eV to 2.58 eV, depending on the deposition conditions.

Keywords Vanadium oxide · Thin films · Optical properties · Spray pyrolysis

1 Introduction

Vanadium oxides are a complex class of materials exhibiting many interesting and novel properties. The main vanadium oxides are VO, V_2O_5 , V_2O_3 , V_4O_9 and VO_2 . V_2O_5 is the most stable with the highest oxidation state [1, 2], having many interesting properties including multi-valance, layered structure, wide optical band gap (2.44 eV), good chemical and thermal stability, excellent thermoelectric and electrochromic properties. Therefore, vanadium oxide thin films and, specially V_2O_5 have been used in fabricating many solid state devices, such as high capacity lithium batteries, display systems, color filters, catalysts, smart windows and gas sensor [3, 4]. In fabricating thin films of materials, the deposition parameters play a significant role in the microscopic and macroscopic properties. Therefore, it is very important to choose the proper deposition parameters for preparing vanadium oxide films for a certain application [5].

Vanadium oxide thin films have been prepared by various methods including pulsed laser deposition (PLD) [6], electron beam evaporation [7], magnetron sputtering [8, 9], sol gel process and spray pyrolysis (SP) [3, 10]. The method of preparation and deposition parameters affect the properties of the prepared samples. Akl [11] studied the effect of thermal annealing on optical properties of the sprayed films of V_2O_5 . In addition, Luo et al. [1] investigated the influence of substrate temperature on the electrical and optical properties of the sputtered V_2O_5 films.

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Among the different deposition techniques for preparing vanadium oxide thin films, the spray pyrolysis (SP) is a relatively simple and inexpensive technique for large-area coatings. However, few efforts have been made to systematically investigate the effects of deposition parameters on the structural, electrical and optical properties of the vanadium oxide thin films fabricated by SP.

In this work, we have systematically studied the effect of the deposition parameters, such as substrate temperature, solution concentration and the solution spray rate on the physical properties of the vanadium oxide films prepared using SP method. The prepared samples were characterized and the measurements were carried out using X-ray diffraction (XRD), scanning electron microscopy (SEM), Hall effect and UV-Vis spectroscopy techniques.

2 Experimental methods

2.1 Film deposition

Vanadium oxide thin films were deposited on glass substrate by SP technique. First, certain amount of vanadium chloride (VCl_3 , $2\text{H}_2\text{O}$) was dissolved in distilled water to make the initial (precursor) solution. All the samples were deposited under a constant nozzle to substrate distance, $d = 35$ cm and compressed air of 3.03×10^5 Pa were used as the carrier.

In the first stage, samples were prepared at different substrate temperatures of (T_{sub}) 300, 400, 450 and 500 °C with constant solution concentration and solution spray rate of 0.1 mol/L (VCl_3 , $2\text{H}_2\text{O}$) and 10 mL/min, respectively.

Fig. 1 XRD patterns of vanadium oxide films **a** prepared at different substrate temperatures T_{sub} and constant vanadium concentration, 0.1 mol/L, and solution spray rate of 10 mL/min **b** deposited at different solution concentrations at $T_{\text{sub}} = 450$ °C and 10 mL/min solution spray rate **c** deposited at various solution spray rates at $T_{\text{sub}} = 450$ °C and solution concentration 0.1 mol/L

Table 1 Summary of XRD parameters and mean grain size of all samples for (200) orientation

	Sample	2 θ (°)	Lattice distance/nm	FWHM/(°)	Mean grain size/nm	Identification with (hkl) value
The effect of different T_{sub} (vanadium concentration 0.1 mol/L and solution spray rate 10 mL/min)	300 °C	–	–	–	–	–
	400 °C	21.52	0.4125	0.301	27.135	Tetragonal V ₄ O ₉
	450 °C	21.53	0.4123	0.293	27.660	Tetragonal V ₄ O ₉
	500 °C	–	–	–	–	–
The effect of vanadium concentration (T_{sub} = 450 °C, solution spray rate 10 mL/min)	0.05 mol/L	21.52	0.4125	0.296	27.350	Tetragonal V ₄ O ₉
	0.10 mol/L	21.53	0.4123	0.293	27.660	Tetragonal V ₄ O ₉
	0.15 mol/L	12.19	0.725	0.343	23.310	Tetragonal β - V ₂ O ₅
	0.20 mol/L	12.72	0.695	0.399	20.210	Tetragonal β - V ₂ O ₅
The effect of solution spray rate (T_{sub} = 450 °C, vanadium concentration 0.1 mol/L)	3 mL/min	12.67	0.6971	0.388	20.810	Tetragonal β - V ₂ O ₅
	5 mL/min	12.71	0.6956	0.327	24.470	Tetragonal β - V ₂ O ₅
	7 mL/min	12.23	0.7220	0.335	24.030	Tetragonal β - V ₂ O ₅
	10 mL/min	21.53	0.4123	0.293	27.660	Tetragonal V ₄ O ₉

The second group of samples were prepared with different solution concentrations, from 0.05 mol/L to 0.2 mol/L, at constant substrate temperature $T_{\text{sub}} = 450$ °C and solution spray rate (10 mL/min). In the final stage, T_{sub} and solution concentration were kept at 450 °C and 0.1 mol/L, respectively, and the solution spray rate was changed from 3 mL/min to 10 mL/min.

2.2 Characterizations

To study the structure of the prepared films, XRD patterns were recorded using D8 Advance Bruker system using Cu K_{α} ($\lambda = 0.15406$ nm) radiation. The average crystallites size (D) of the samples was estimated using the Scherrer's formula [12]:

$$D = \frac{k\lambda}{\delta_w \cos \theta}, \quad (1)$$

where δ_w is the full width at half maximum (FWHM) of the corresponding XRD peak, k a constant (about 0.9) and θ the Bragg angle. Surface morphology of the films was studied using Philips XL-30 SEM system. The type and concentration of the majority charge carriers were determined by Hall effect experiment. The majority charge carrier concentration was obtained using the following equation [12]:

$$N_{n,p} = \frac{IB}{|q|V_H t}, \quad (2)$$

where I , B , q , V_H and t are the electric current, magnetic flux density, electron charge, Hall voltage and film thickness, respectively.

Optical measurements were carried out in the range of 190–1,100 nm wave lengths, using Unico 4802 spectrophotometer system. The direct band gap (E_g) of the

prepared films was obtained from the extrapolation of the linear part of the $(\alpha h\nu)^2$ curve versus photon energy ($h\nu$) using the following equation [13]:

$$(\alpha h\nu)^2 = A(h\nu - E_g), \quad (3)$$

where α and E_g are the absorption coefficient and the energy band gap, respectively, and A is a constant. The extinction coefficient of the prepared vanadium oxide films was calculated by the following relation [14]:

$$k = \frac{\alpha \lambda}{4\pi}, \quad (4)$$

where α and λ are the absorption coefficient and wavelength, respectively.

The sheet resistance (R_s) of the films was measured by two-point probe method with Al electrodes made by thermal evaporation in vacuum using Edwards E-306 coating system.

3 Results and discussions

3.1 Structural properties

Figures 1a–1c show the XRD patterns of the vanadium oxide films prepared at different T_{sub} , solution concentrations and solution spray rates.

Figure 1a shows the XRD patterns of the samples prepared at T_{sub} of 300, 400, 450 and 500 °C, keeping vanadium concentration and solution spray rate at 0.1 mol/L and 10 mL/min, respectively. The patterns show that the structure of the films prepared at 300 °C and 500 °C are almost amorphous with a low intensity peak at $2\theta = 31.8^\circ$ which can be related to V₃O₇ phase, but the samples made at 400 °C and 450 °C have tetragonal V₄O₉ structure with the preferred orientation along

Fig. 2 SEM images of vanadium oxide films prepared at **a** $T_{\text{sub}} = 300$ °C **b** $T_{\text{sub}} = 500$ °C with vanadium concentration 0.1 mol/L and solution spray rate 10 mL/min, deposited in **c** 0.05 mol/L **d** 0.2 mol/L at $T_{\text{sub}} = 450$ °C and 10 mL/min rate, deposited at **e** 3 mL/min **f** 7 mL/min solution spray rate at $T_{\text{sub}} = 450$ °C and vanadium concentration 0.1 mol/L

Fig. 2 continued

(200) direction. The intensity of the (200) peak decreases with increasing the T_{sub} from 400 °C to 450 °C, along with appearing β - V_2O_5 phase with tetragonal crystallographic lattice along (200) direction. The formation of the polycrystalline films at 400 °C and 450 °C can be due to the complete chemical reactions leading to the higher lattice order.

Figure 1b shows the XRD patterns of vanadium oxide films prepared at constant $T_{\text{sub}} = 450$ °C and solution spray rate of 10 mL/min with different vanadium concentrations. The patterns show that all the prepared samples contain both β - V_2O_5 and V_4O_9 phases, but the prepared films with 0.2 mol/L give the single phase of β - V_2O_5 with orientation along (200) direction. In addition, with increasing the solution concentration from 0.05 mol/L to 0.2 mol/L, the intensity of the preferred orientation along (200) of V_4O_9 has decreases but that of (200) peak corresponding to β - V_2O_5 has increases.

Figure 1c shows the XRD patterns of the films prepared with various solution spray rates, while keeping T_{sub} and vanadium concentration at 450 °C and 0.1 mol/L. All the samples have β - V_2O_5 phase and with increasing the solution rate from 3 mL/min to 5 mL/min, the intensity of the preferred orientation along (200) is increased. The intensity of (200) peak for the rate of 5 mL/min is very strong, hence it can be deduced that the structure is nearly single crystal. By increasing the solution rate from 5 mL/min to 10 mL/min, the intensity of the (200) peak decreases. The V_4O_9 phase appears in the samples

prepared with 7 mL/min and 10 mL/min along (200) orientation. The information obtained from the XRD analysis is summarized in Table 1.

SEM images of the samples prepared under different conditions are given in Figs. 2a–2f. Figures 2a and 2b are the SEM images of the sample prepared at $T_{\text{sub}} = 300$ °C and $T_{\text{sub}} = 500$ °C with vanadium concentration 0.1 mol/L and constant rate 10 mL/min. It can be seen from Figs. 2a and 2b that the crystallization has been completed by increasing T_{sub} from 300 °C to 500 °C and nano needles shape of the grains are formed.

Figures 2c and 2d show the SEM images of the sample made with vanadium concentration 0.05 mol/L and 0.2 mol/L at $T_{\text{sub}} = 450$ °C with the solution spray rate 10 mL/min. The figures show that by increasing the vanadium concentration from 0.05 mol/L to 0.2 mol/L the needle shape structures are formed. In addition, in Figs. 2e and 2f, it is clear that the structure of the deposited vanadium oxide thin film is much better for the lower rate of spray (3 mL/min) than the one with 7 mL/min.

3.2 Electrical properties

The results of the electrical measurements of the films prepared at different T_{sub} , solution concentrations and solution spray rates are summarized in Table 2. The thickness of the films, which were determined from the transmission data using the Puma software [15] are also given in Table 2.

Table 2 Electrical measurement results (thickness determination error is about $\pm 5\%$)

	Sample	$R_s/(\text{M}\Omega)$	Carrier type and carrier concentration/ cm^{-1}	Thickness t/nm
The effect of different T_{sub} (vanadium concentration 0.1 mol/L, solution spray rate 10 mL/min)	300 °C	1.27	0.660×10^{16} (+)	210
	400 °C	0.77	0.550×10^{16} (−)	200
	450 °C	0.60	0.630×10^{16} (+)	220
	500 °C	2.50	0.260×10^{16} (−)	250
The effect of vanadium concentration ($T_{\text{sub}} = 450$ °C, solution spray rate 10 mL/min)	0.05 mol/L	0.550	0.135×10^{18} (−)	230
	0.10 mol/L	0.600	0.630×10^{16} (+)	220
	0.15 mol/L	0.597	0.157×10^{18} (−)	240
	0.20 mol/L	1.160	0.250×10^{16} (−)	350
The effect of solution spray rate ($T_{\text{sub}} = 450$ °C, vanadium concentration 0.1 mol/L)	3 mL/min	6.610	0.100×10^{16} (−)	240
	5 mL/min	0.940	0.119×10^{17} (−)	350
	7 mL/min	0.610	0.740×10^{17} (−)	350
	10 mL/min	0.597	0.630×10^{16} (+)	240

Fig. 3 Transmission of the samples **a** prepared at different substrate temperatures with constant vanadium concentration 0.1 mol/L, deposited with **b** different precursor solutions at $T_{\text{sub}} = 450$ °C and 10 mL/min solution spray rate, and **c** various solution spray rates at $T_{\text{sub}} = 450$ °C and vanadium concentration 0.1 mol/L

Fig. 4 Extinction coefficient of vanadium oxide films **a** prepared at different T_{sub} with vanadium concentration 0.1 mol/L, deposited with **b** different precursor solutions at $T_{\text{sub}} = 450$ °C and solution spray rate 10 mL/min rate and **c** various solution spray rates at $T_{\text{sub}} = 450$ °C and vanadium concentration 0.1 mol/L

The electrical measurements reveal that the R_s of the films prepared at different T_{sub} with constant vanadium concentration (0.1 mol/L) and the solution spray rate (10 mL/min) decreases with increasing T_{sub} , due to the growth of the grain size and the improvement of the stoichiometry of the samples shown by XRD patterns. At $T_{\text{sub}} = 500$ °C, the R_s of the samples increases, due to the decrease of the charge carrier concentrations. The R_s of the samples prepared with different solution concentrations at $T_{\text{sub}} = 450$ °C and solution spray rate 10 mL/min is found to increase with increasing the solution concentration. This is related to the decrease of the charge carrier concentrations or decrease of the grain size. The R_s of the samples prepared with various solution spray rates and constant vanadium concentration of 0.1 mol/L and $T_{\text{sub}} = 450$ °C decreases with increasing the solution rate, because of the increase of the grains size and the charge carrier concentration of the films. The lowest R_s is obtained for the films prepared at $T_{\text{sub}} = 450$ °C, vanadium concentration 0.05 mol/L and spray solution rate 10 mL/min. The

Hall effect experiment shows that in all samples the majority carriers are electrons (*n*-type conduction) except for the samples prepared at $T_{\text{sub}} = 300$ °C and 450 °C with 0.1 mol/L vanadium concentration and 10 mL/min solution spray rate which are *p*-type.

3.3 Optical properties

The transmittance spectra of the prepared films are shown in Figs. 3a–3c. The spectra in Fig. 3a show that by increasing the T_{sub} from 400 °C to 500 °C, at constant 0.1 mol/L vanadium concentration and solution spray rate (10 mL/min), the transmittance increases from 20% to 60%. Figure 3b shows the optical transmittance spectra of the samples prepared, at constant $T_{\text{sub}} = 450$ °C and the solution spray rate 10 mL/min, with different VCl_3 molarities. The transparency of the films decreases from 52% to 41% with changing the molarity from 0.05 mol/L to 0.1 mol/L. This decrease may be due to the density or non-homogeneity

Fig. 5 $(\alpha h\nu)^2$ versus $h\nu$ (photon energy) for **a** films prepared at different T_{sub} with vanadium concentration 0.1 mol/L and solution spray rate 10 mL/min, **b** deposited films with various precursor solutions at $T_{\text{sub}} = 450$ °C and solution spray rate 10 mL/min, **c** the films deposited with various solution spray rates at $T_{\text{sub}} = 450$ °C and vanadium concentration 0.1 mol/L

of the films which is more for the sample prepared with 0.1 mol/L than the sample prepared with 0.05 mol/L due to the increase of the reflected lights and therefore the decrease of the transmittance of the film. The transmittances of the films prepared with 0.15 mol/L and 0.2 mol/L vanadium concentrations are almost the same and are about 47% which is higher than for the film made with 0.1 mol/L. This can be related to the oxidation level of the sample. The transmission spectra of the films prepared at $T_{\text{sub}} = 450$ °C and vanadium concentration of 0.1 mol/L but with different solution spray rates are shown in Fig. 3c. It is found that the transmittance decreases from 75% to 41%, when spray rate is changed from 3 mL/min to 10 mL/min. This decrease, for the rates from 3 mL/min to 7 mL/min, is due to the increase of the film thickness but for 10 mL/min the film thickness is decreased (see Table 2). The formation of amorphous phase or decrease of oxidation level is the major cause for the decrease of optical transmittance.

The calculated extinction coefficient curves, k versus λ , of the prepared samples are given in Figs. 4a–4c. In all samples, k decreases with increasing the wavelength in the

range 400–600 nm. The value of k decreases from 0.8 to 0.23 with increasing the T_{sub} and from 0.55 to 0.3 with increasing the molarity of starting solution (VCl_3) at constant solution spray rate of 10 mL/min. The value of k for the samples made with different solution spray rates and constant vanadium concentration of 0.1 mol/L and $T_{\text{sub}} = 450$ °C increases from 0.23 to 0.4 with increasing the solution spray rates from 3 mL/min to 10 mL/min. The increase of k , corresponding to the decrease of the transmittance, occurs at short wavelengths due to the increase of the conductivity of the films. The value of the band gap of the samples was obtained as depicted in Figs. 5a–5c. As shown in Fig. 5a, by increasing the T_{sub} from 300 °C to 500 °C, at constant vanadium concentration 0.1 mol/L and the solution spray rate 10 mL/min, the obtained energy band gap value E_g reduces from 2.46 eV to 2.22 eV, which is related to the grain size growth and/or multi phase nature of the films [16]. Figure 5b reveals that by increasing vanadium concentration from 0.1 mol/L to 0.2 mol/L at $T_{\text{sub}} = 450$ °C and the solution spray rate 10 mL/min, the E_g is increased slightly from 2.36 eV to 2.4 eV because of

the decrease of grain size. However, the deposited film at vanadium concentration 0.05 mol/L has a band gap of 2.46 eV. By increasing the solution spray rate from 3 mL/min to 10 mL/min with constant vanadium concentration 0.1 mol/L and $T_{\text{sub}} = 450\text{ }^{\circ}\text{C}$, there is a decrease in E_g (from 2.58 eV to 2.36 eV), as shown in Fig. 5c, which can be related to the increase of the grain size of the layers.

4 Conclusions

In this paper, preparation and characterization of vanadium oxide thin films deposited on glass substrates SP technique are reported. The effects of different T_{sub} , vanadium concentrations in precursor solution and the spray rates on the physical properties of the prepared samples have been investigated. The results revealed that the physical properties of the films depend strongly on the deposition conditions. By analyzing the results, one can conclude the followings:

- (i) The structure of the deposited films are mostly tetragonal with $\beta\text{-V}_2\text{O}_5$ and V_4O_9 phases having the preferential growth along (200) direction. By increasing T_{sub} from $300\text{ }^{\circ}\text{C}$ to $400\text{ }^{\circ}\text{C}$, V_4O_9 phase is observed along (200) direction but with further increasing of the temperature the intensity of V_4O_9 phase reduces and $\beta\text{-V}_2\text{O}_5$ phase appears. In addition, it is revealed that the crystallization is better formed at higher T_{sub} . Increasing of vanadium concentration causes the decrease of V_4O_9 and the increase of $\beta\text{-V}_2\text{O}_5$ intensity. In the samples prepared with different solution spray rates, the intensity of $\beta\text{-V}_2\text{O}_5$ phase decreases and V_4O_9 phase appears in the structure. The solution spray rate of 5 mL/min was found to be the optimum value which gives almost the single crystal of $\beta\text{-V}_2\text{O}_5$.
- (ii) The electrical measurements show that the R_s of the prepared films decreases with increasing the T_{sub} up to $450\text{ }^{\circ}\text{C}$, but at $500\text{ }^{\circ}\text{C}$ increases sharply. However, the R_s decreases with increasing the solution spray rate, and increases with increasing vanadium concentration. From Hall effect measurements, it was found that all the samples were n -type except the films deposited at $T_{\text{sub}} = 300\text{ }^{\circ}\text{C}$ and $450\text{ }^{\circ}\text{C}$ with 0.1 mol/L vanadium concentration and 10 mL/min solution spray rate which showed p -type behavior.
- (iii) The transparency of the films deposited at different R_s increased from 20% to 60% with increasing temperature from $400\text{ }^{\circ}\text{C}$ to $500\text{ }^{\circ}\text{C}$. The transparency of the films at different solution concentrations decreases from 52% to 41% with increasing the morality of the starting solution.

Increasing the spray rate causes the decreasing in the transparency of the films. By increasing the T_{sub} and the solution spray rate, the E_g of the samples decreases gradually from 2.46 eV to 2.22 eV and from 2.58 eV to 2.36 eV, respectively. By increasing VCl_3 concentration, the E_g increased from 2.36 eV to 2.4 eV. However, for vanadium concentration of 0.05 mol/L, $T_{\text{sub}} = 450\text{ }^{\circ}\text{C}$ and the solution spray rate of 10 mL/min, the obtained E_g is 2.46 eV.

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