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Preparation and Characterization of Vanadium Pentoxide Using Spray Pyrolysis Technique

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Abstract

A chemical spray pyrolysis technique was used to deposit a vanadium pentoxide (V_2O_5) thin films on glass substrate with a deposition temperature ranged from 300°C to 500°C in step of 50°C. From ammonium meta vanadate aqua precursor solution molarity of (0.1 M) was used as a source of vanadium. The effect of deposition temperature on structure, morphological, electrical conductivity and optical properties wasanalyzedat constant preparation time, solution molarity and the distance between spray nozzle and substrate.X-ray diffraction patterns shown that an orthorhombic cubic structure withgrowth along (001) plane. With increasing the substrate temperature, the electrical conductivity was increased, and the scanning electron microscopy clarified that the crystallinity of V_2O_5 thin films was effectively modified. The optical results revealed that energy band gap of V_2O_5 films deposited at 400^{o}C, 450^{o}C and 500^{o}C is 2.38 eV for direct allowed transition. Based on the observed results the V_2O_5 phase can be well controlled by altering the substrate temperature. All prepared thin films up to 400^{o}C show transparency in both visible and near IR region.

Keywords: V_2O_5, XRD, Morphological, Optical, Electrical properties, Depositiontemperature, Crystallinity andgraingrowth.

1.Introduction

Among the transitions metal oxide semiconductors, vanadiumpentoxide, particularly in thin-film structure, takes concerned widely through many years because of its varied range of uses [1,9]. Multi-valance layered construction characterized by wide-ranging band-gap, respectable stability of chemical and thermal properties. The outstanding thermo-electric feature that is the character which sort a vanadium pentoxide

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(V_2O_5) hopeful material for micro-electronics, electro-chemical, and optic-electronic devices [2-5]. Recent researches studies have pointed out that with the application of electrical signal [40]. Nano fibers made up of vanadium oxide can act as vicarious muscles that can contract [9]. Many techniques such as pulsed laser deposition (PLD) [10] gives a brief overview of the progress that it has made starting with control of deposition parameters such as a deposition temperature [36,37]. Sol-gel spin coating [11] where that an orthorhombic structured thin film is transformed to β-(V_2O_5) nanorods by subsequent annealing at 500 °C. The as-deposited V_2O_5thin films were produced by thermal evaporation technique [12] without intentional substrate heating present an amorphous structure. After thermal treatment for 1h at atmospheric environment conditions the films show a predominant (001) plane reflection of the orthorhombic V_2O_5 phase. Direct current radio frequency (DC/RF) sputtering [13] was studied the influence of ambient atmospheres on the structure, optical properties,and morphology of the thin films after annealing. The characterization and the transition behaviors in the annealing process were investigated by the dominant sequence clustering (DSC). The results demonstrated that V_2O_5 films underwent four different transition behaviors during post-deposition annealing due to the different oxygen proportion of ambient. Electron beam evaporation technique [14] showed that SEM micrographs perceived the prepared films were nearly homogeneous with densely packed morphology, from the optical absorption data, the indirect optical band gap (E_g) was found to be 2.36 eV. The reported of [15] using vacuum evaporation method clarified that XRD of films deposited at substrate temperature 350°C were an orthorhombic structure. Moreover, the degree of crystallization is improved by thermal annealing with a preferential orientation along the direction <001 >.

Spray pyrolysis technique [16–25] has been found to be extensively used by the researchers with a variety of oxide materials; it was reported to obtain various forms of vanadium oxides. The reason is attributed to its low-cost setup and its capability to deposit large area and thus it is suitable for substrates with the wide surface-area. This technique also provides the best adherence of the metal oxide thin films [26-28]. V_2O_5 thin films were deposited by the spray pyrolysis method on pre-cleaned glass substrates using ammonium vanadate (NH_4VO_3) as a starting material dissolved in water solvent [6,7], and the deposition temperature-dependent morphological, structural, electrical and

optical properties of it were investigated and reported [29-31]. Where X-ray diffraction studies have shown the thin films were composed of (V_2O_5) with a cubic orthorhombic structure and SEM images showed that the size and shape of the flakes formed on the surface of the film deposited effectively modified with increasing substrate temperature [8, 9]. The effect of annealing on microstructure and optical properties of (V_2O_5) thin were studied.

In this work, vanadium pentoxide (V_2O_5) thin films have been successfully deposited onto preheated glass substrates via chemical spray pyrolysis deposition method at various substrate temperatures from 300 to 500°C. The influence of the substrate temperature on structure, morphological, electrical conductivity and optical properties is discussedto understand the growth process based on the observedexperimental results of V_2O_5 thin films.

2. Experimental

By using spray pyrolysis technique, the thin films of the vanadium pentoxide (V_2O_5) were deposited on glass substrate at different deposition temperature 300,350,400,450 and 500℃ at constant spray time 20 min. This particular time is the most appropriate time in the previous work [32] to obtain the highest quality thin films formed. The solution molarity (0.1 M) of ammonium metavanadate (Segma Aldrich with purity 99.0%) which solved in distilled water used as a precursor [29]. The substrate temperature was kept with accuracy of ±5°C with the help of the feedback control system for the heater supply. The compressed air was used as atomization gas carrier. The atomizer which used is automatic spray gun HM-3 from FUSO SEIKI Co., Ltd.; Japan. In order to get uniform thin films, the distance between the nozzle and the substrate, and pressure of the carrier gas was kept constant for all samples at 35 cm, and 1.5 bar respectively. The glass substrates were carefully cleaned by distilled water, acetone and ethanol; for 10 min in ultrasonic respectively. The X-ray diffraction (XRD) of prepared films was obtained by Philips diffractometer model (PW3040) using $K_{\alpha 1}$radiation (λ= 1.54Å) in range. The optical properties were obtained by UV/Vis/NIR Spectro photometer model Jasco-670 in the wavelength range 300–2500 nm [29]. The morphology of the films was examined by using Scan electronic microscope (FEI Quantum model FEG 250) [30].

The thickness of the prepared films measured by a Stylus profilmeter model

(Dektak 150). Sheet resistance measured by using Keithly Bench meter model 2110 at room temperature by two points configuration.

3. Results and discussion

3.1. Structural properties

Figure 1 shows X-ray diffraction patterns for (V_2O_5) thin-films at different substrate temperatures and a constant spray time of 20 min using origin lab program to draw it. X-ray diffraction patterns showed that the thin layers at 300°C and 350°C were amorphous because the temperature is not sufficient to form (V_2O_5) phase but can form another vanadium oxide phase that is in accordance with results in [26, 33, 39, 41]. Where the deposited films at substrate temperature up to 350 ° C (V_2O_5) phase were formed with an orthorhombic structure of (V_2O_5) phase according to (JC PDS card no. 00-041 - 1426) with the growth orientation along (001) plane positioned. The peaks located at 2θ=20.72,21.8,41.5 were assigned to the (001), (l0l) and (002) reflections, respectively [23]. The value of 2θ located for (001) peak of the deposited thin-films at 400ºC,450ºC, and 500ºCwas increased to higher angle value compare to the standard value of (2θ) 20.26, thus signifying compressive strength in the layers. The intensity of (001) peak increases with increasing the deposition temperature up to 500ºC thus clarify the development in the crystallinity of the prepared thin-films(V_2O_5).

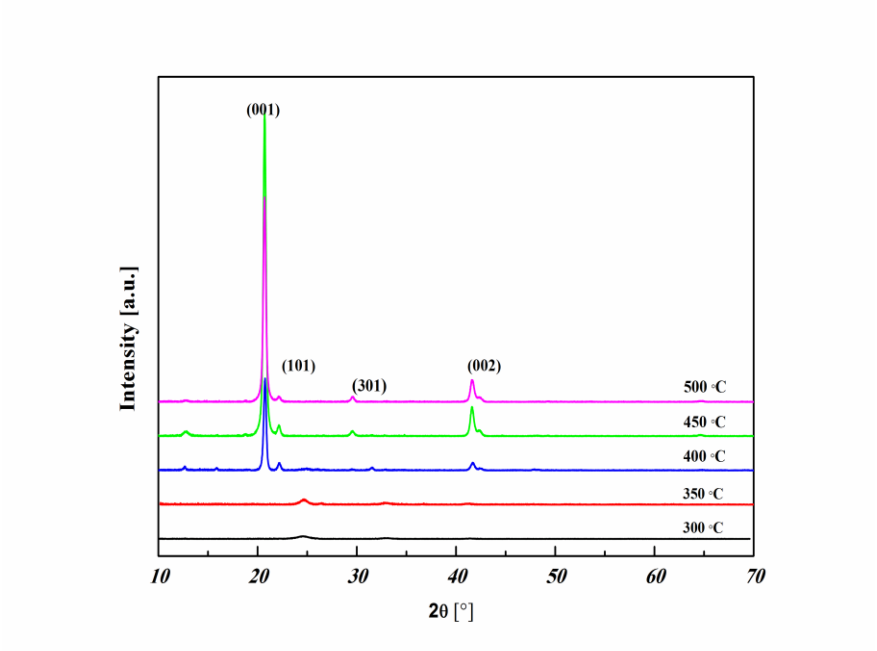


Fig.1 X-ray diffraction patterns of (V_2O_5) thin films at different substrate temperatures.

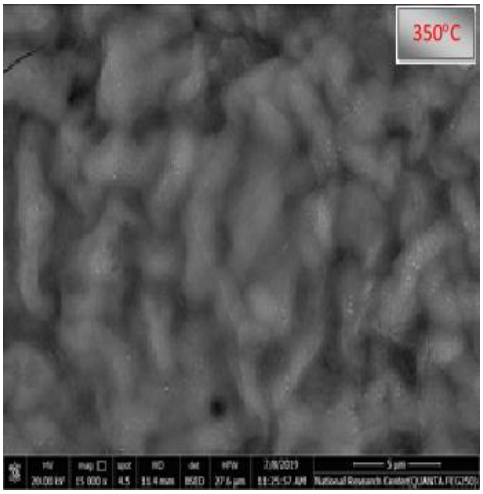
The average crystal size (D) was estimated using Scherer's formula [1]:

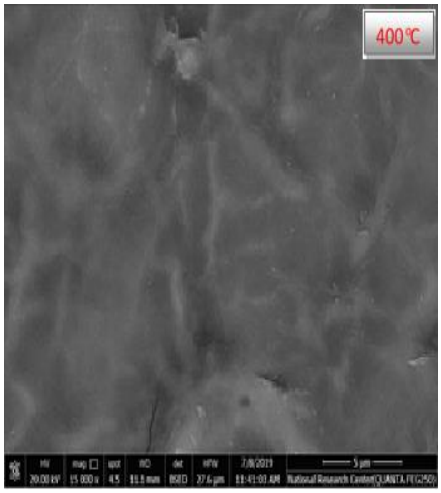
$$D = \frac{0.94\lambda}{\beta\cos\theta} \qquad (1)$$

Where θ is Bragg angle and β is the full width at half maxima of the diffraction peaks and λ=1 .54 A. The structural parameters for (001) crystallographic orientation of the (V_2O_5) thin films shows that with increasing the deposition temperature, the crystallite size increases up to 4.7 nm. It is noted that larger crystallites were formed at substrate temperatures of 400ºC, 450ºC and 500ºC for the (001) reflections. The results were in agreement with the previously published [23].

3.2. Morphologicalproperties

Fig.2 Show the SEM images of the (V_2O_5), thin films were deposited at various substrate temperatures 350, 400, 450, and 500°C. The surface property of the (V_2O_5) films was changed as a function of substrate temperature. The grain growth of (V_2O_5), was found to be enhanced with increasing substrate temperature. Fig.2. Shows that with increasing deposition temperature, the small clusters were merged with each other to form bigger grains and the (V_2O_5) films become more compact, dense and adhered to the entire substrate without any cracks this result was agree with [20,21,24,33].





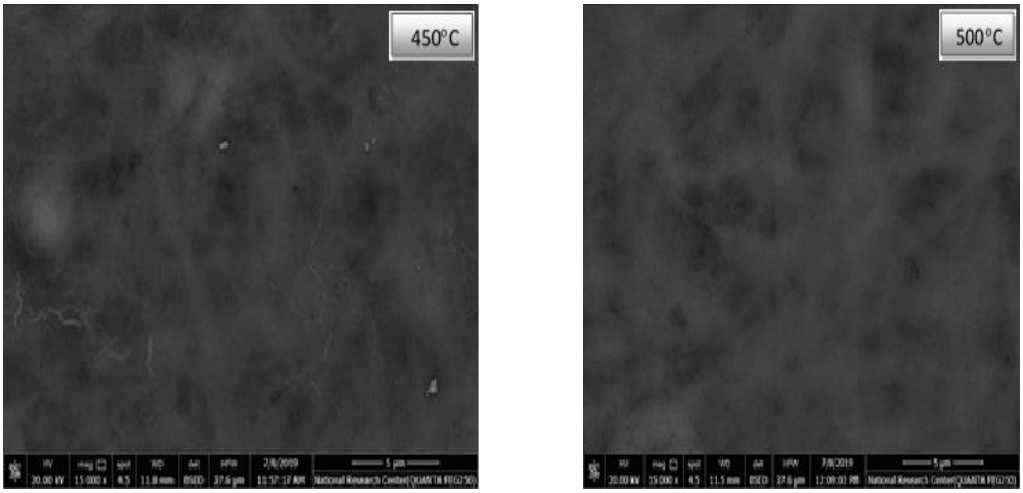


Fig.2. SEM imagesof (V_2O_5) thin films sprayedat different substrate temperatures.

3.3. Electrical properties

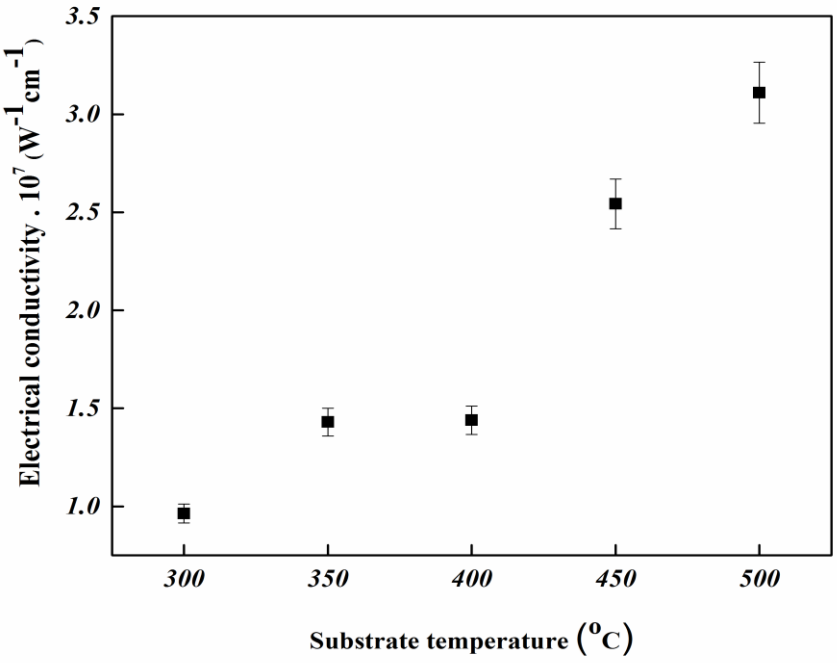


Fig.3 Electrical conductivityof (V_2O_5) thinfilmdepositedatdifferenttemperatures.

The DC electrical conductivity of (V_2O_5)thin-films prepared at different substrate temperature was measured using two-probe configuration. Fig.3 Shows the variation of electrical conductivity with substrate temperature. With increasing the substrate temperature, the electrical conductivity increased, which may be attributed to the enhanced crystallinity and grain growth of the films with increasing the substrate temperature. These

results were confirmed by XRD and SEM data [35].

3.4. Optical properties

At various substrate temperatures the (V_2O_5) thin films optical transmittance spectra was all set shown in Fig.4 All prepared thin-films up to 400°C show transparency in both visible and near IR regions. The optical transparent increased by increasing the substrate temperature, this enhancing in the transparency can be attributed to the enhancing of the crystallinity and crystallite size [34].

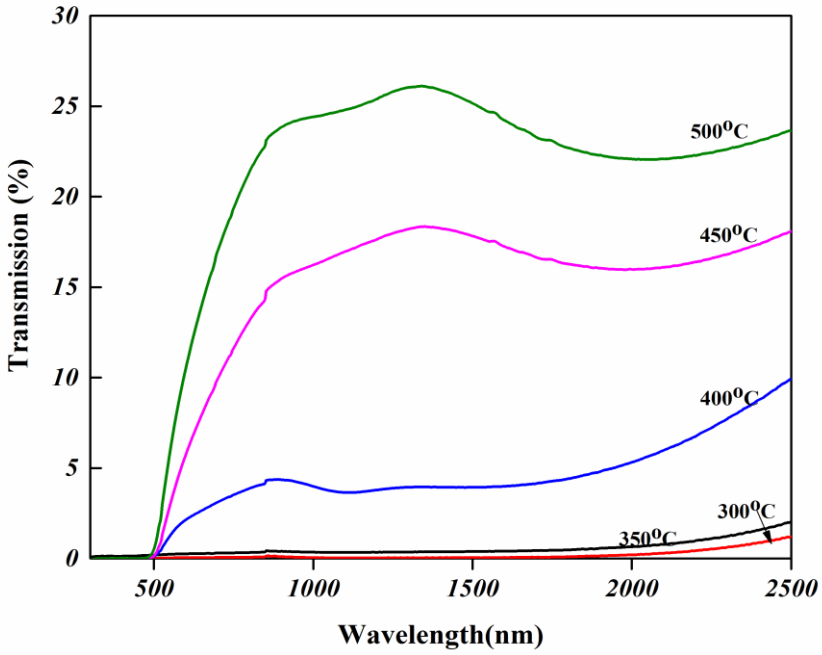


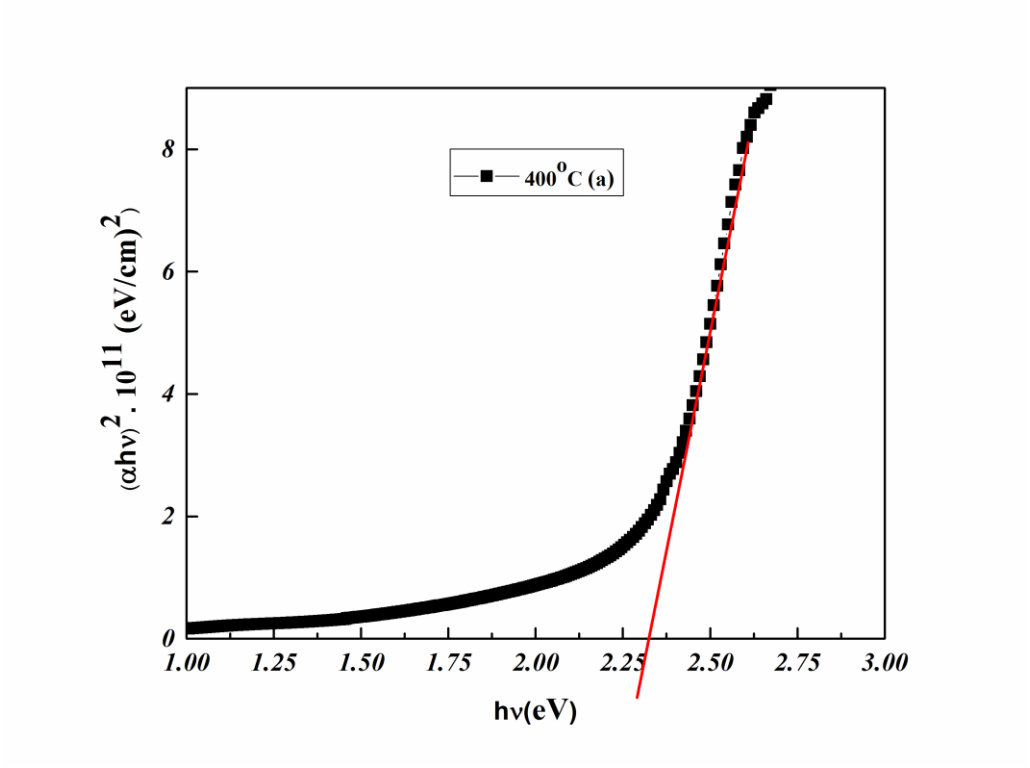
Fig.4Opticaltransmissionspectrum of (V_2O_5) thin films deposited at different substrate temperatures.

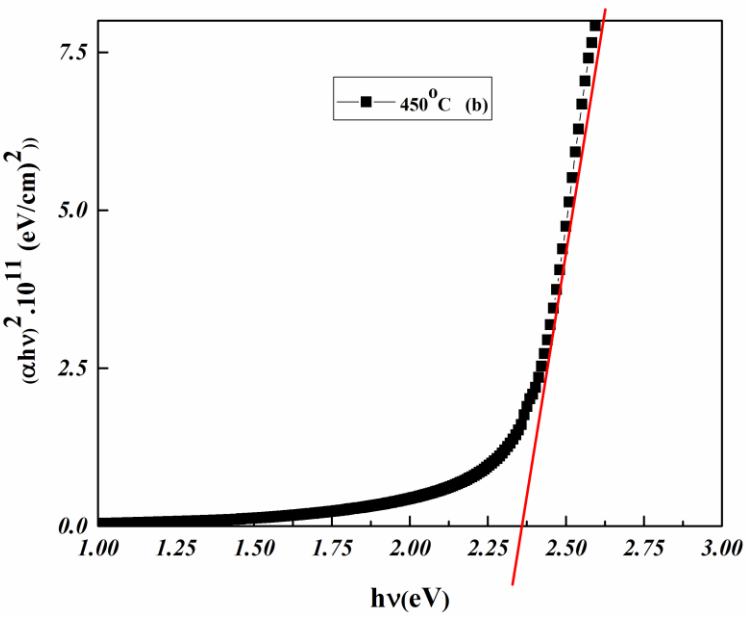
The E_g value can be calculated using the fundament absorption, when the electron excited from the valance band to conduction band. The extrapolations of the linear portion of the data curve between $((\alpha h\upsilon)^2 vs h\upsilon)$ measured the (E_g) value, as shown in Fig.5. Wherethe photon energy is ($h\upsilon$).

The absorption coefficient (α) is expressed as:

$$\alpha = \frac{\ln(\frac{1}{T})}{d} \qquad (2)$$

Where T is transmittance and d is film thickness.


400°C (a)
(αhν)2 . 1011 (eV/cm)2)
hν(eV)
0
2
4
6
8
1.00
1.25
1.50
1.75
2.00
2.25
2.50
2.75
3.00


450°C (b)
(αhν)2.1011 (eV/cm)2))
hν(eV)
0.0
2.5
5.0
7.5
1.00
1.25
1.50
1.75
2.00
2.25
2.50
2.75
3.00

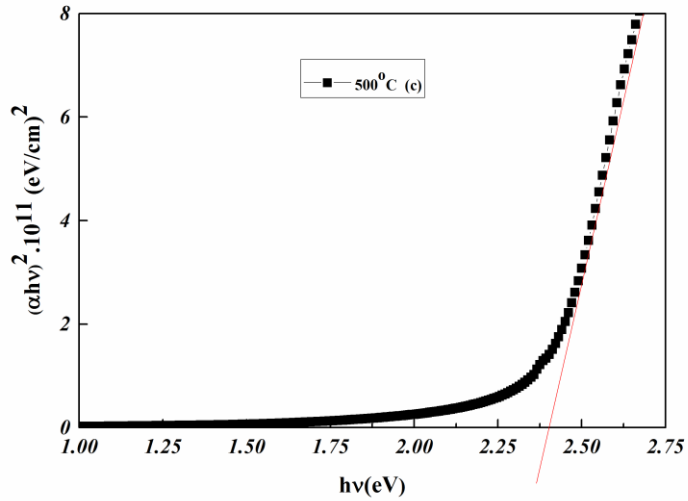


Fig.5 (a, b, c) relation between $(\alpha h\upsilon)^2$ and hυ for (V_2O_5) thin films prepared at different substrate temperatures.

The absorption coefficient and the incident photon energy (hυ) relation were calculated by equation (3),

$$(\alpha h\upsilon)^2 = A(h\upsilon - E_g) \qquad (3)$$

Where A is a constant and E_g is optical band-gap.

The energy band gap of the prepared samples at substrate temperature 400°C,450°C and 500°C was almost having the same value 2.38 eV. This value is corresponding to (V_2O_5) phase bulk (V_2O_5) value of Eg~ 2.2 eV [27-30]. The change in the growth behavior and morphology of the sprayed (V_2O_5) films with changing in the substrate temperature may because a small variation in the optical band gap [38].

4. Conclusion

By using a spray pyrolysis technique, a conducting and transparent (V_2O_5) thin films were successfully deposited on a cut-glass substrate. The influence of the substrate temperatures on structural, morphological, electrical and optical properties of (V_2O_5) films was investigated. X-ray diffraction patterns revealed that orthorhombic (V_2O_5) phase withcrystalstructure begin to form at deposition temperature of 400°C to 500°C. The crystalline size of (V_2O_5) was found to be enhanced with increasing substrate temperatures. The optical transmittance in the visible and near infra-red regions increasing with increasing

the substrate temperatures. The energy band gap of (V_2O_5) samples is 2.38 eV for all substrate temperatures. The films deposited at 500°C have the highest conductivity value $3x10^3\ \Omega^{-1}cm^{-1}$.

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الملخص العربي

تحضير وتوصيف خامس أكسيد الفاناديوم باستخدام تقنية الانحلال الحراري للرذاذ

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2- قسم الفيزياء كليه البنات للاداب والعلوم والتربية جامعه عين شمس هليوبلس القاهره.

3-قسم فيزياء الجوامد المركز القومي للبحوث الرقم البريدي 12311الدقي الجيزه.

استخدمت تقنية الانحلال الكيميائي الحراري للرذاذ لترسيب أغشية رقيقة من خماسي أكسيد الفاناديوم(V_2O_5)على شريحة زجاجية في مدي من درجات الحرارة 300 إلى 500 درجة مئوية كل 50 درجة مئوية باستخدام محلول مائى من ميتا فانادات الأمونيوم بتركيزمولى (0.1 مول). تم تحليل وتسجيل تأثير درجة حرارة الترسيب على الخصائص الهيكلية السطحية،الموصلية الكهربائية،والخصائص الضوئية في ثبوت بقية العوامل الأخرى مثل وقت تحضير أغشية رقيقة من خماسي أكسيد الفاناديوم(V_2O_5)، ومولارية المحلول، والمسافة بين فوهة الرش والشريحة. أظهرت أنماط حيود الأشعة السينية ان الطور المعيني مع بنية بلورية مكعبة الشكل يزداد في اتجاه المستوي البلوري(001). وأن الحجم البلوري يزداد حتى 4.7 نانومتر بزيادة درجة حرارة الركيزة.كما أوضحت صور المجهر الإلكتروني الماسح أن زيادة درجه حرارة الشريحة يؤدي الي زيادة التبلور ونمو الحبيبات في أغشية خماسي اكسيد الفاناديوم (V_2O_5). كما أوضحت دراسة الخواص الضوئية أن جميع الأغشية الرقيقة المعدة في درجات حرارة اعلي من 400 درجة مئوية تظهر الشفافية في كل من مناطق الأشعة تحت الحمراء المرئية والقريبة. وفجوة نطاق الطاقة للعينات المعدة عند درجة حرارة الركيزة 400و450و500 درجة مئوية لها نفس القيمة تقريبا (2.38) إلكترون فولت. كذلك اوضحت القياسات الكهربية أن الموصلية الكهربائية تزداد بزيادة درجة الحرارة.