Impact of Substrate Temperatures On the Properties of V$_2$O$_5$ Thin Films Deposited by Pulsed Laser Deposition

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Abstract. Vanadium pentoxide (V$_2$O$_5$) thin films were fabricated by pulsed laser deposition (PLD) on fused silica substrate at temperatures ($T_s$) ranged from ambient temperature up to 300°C. UV-VIS-NIR spectral measurements, X-ray diffraction (XRD) X-ray photoelectron spectroscopy (XPS), and scanning electron microscopy (SEM) were made to understand the influence of substrate temperature on optical, structural, and compositional properties. The substrate temperature displayed a robust effect on the construction and visual characteristics. The photosensitive band gap of PLD V$_2$O$_5$ films was powerfully dependent on the substrate temperature and was reduced from 2.36 eV to 2.08 eV with the growth of substrate temperature from ambient temperature to 300°C. However, the refractive index showed an increase from 2.28 to 2.69 for the same temperature range. V$_2$O$_5$ films grown at $T_s = 300°C$ exhibited a crystalline nature as evidenced by XRD and SEM studies. The chemical composition of V$_2$O$_5$ films has been studied by XPS and the data revealed pure V$_2$O$_5$ compound was formed.

Keywords: Vanadium oxide pulsed laser deposition, substrate temperatures, grain size, structure, compositional and optical properties.

1. Introduction

Vanadium available in several oxide forms (VO$_2$, V$_2$O$_3$, and V$_2$O$_5$). Some researchers have been managed in thin-film system and functional as visual and electrical strategies. As a wide bandgap and n-type semiconductor material, vanadium pentoxide (V$_2$O$_5$) is particularly useful. Because of its intriguing electrical conductivity, it has received a lot of attention. performance [1, 2], and integration in lithium auxiliary batteries[3]. V$_2$O$_5$ has been extensively considered in recent years into many scientific and technological applications[4]. Electrochromic materials, digital data demonstrations, and colour storage systems all have a lot of potential with V$_2$O$_5$ [5, 6]. Due to their ability to incorporate vast quantities of lithium ions mixed with their peculiar optical properties, vanadium oxides have some of the most studied materials for electrochemical applications in recent years, and in general and especially for applications that require high energy density solid-state batteries and information displays. V$_2$O$_5$ films' variable optical properties are used in the development of smart devices. Chemical detecting, photochromic, catalysis, and optical and electrical switching are just a few of the functions that V$_2$O$_5$films can perform in other technological applications. [7-10]. Because the processing window during which these oxides exist as a stable single-phase material is so small, tuning the process parameters for controlled growth and desired properties is one of the main difficulties met during the preparation of vanadium oxides in thin-film system. Vanadium procedures a variety of oxides, each of which is steady above a wide range of compositions [11-14].
configuration and phase constancy of full-grown films is extremely significant for all applied requests.

$V_2O_5$ films can be gotten by thermal vanishing[15], flash fading[16], electron-beam vaporisation[17], sol-gel evolution procedures[18], chemical steam confession, and spitting. In the last decade, extensive and successful efforts have been made for thin-film processing of vanadium oxides using pulsed laser deposition because it is an appealing choice for the preparation of stoichiometric and high-quality metal oxide films (PLD) [19-21]. PLD is a versatile and influential performance that has been effectively used to deposit a wide range of materials in the past [21-27]. In this paper, the impact of the substrate temperature on the compositional, optical, and structural properties of $V_2O_5$ films equipped by pulsed laser deposition was studied. The results obtained from UV-Vis-NIR spectra, SEM, XRD and XPS spectroscopies are presented and connected with different substrate temperatures controlled during deposition.

2. Experimental details

Pulsed laser deposition with Lambda Complex 201 excimer laser ($\lambda = 308$ nm) with the influence of $2.55 \text{ Jcm}^{-2}$ was used to fabricate vanadium oxide thin films on fused silica substrates. The rotating target used was a pure ceramic $V_2O_5$. The vacuum chamber was impelled to a base compression of $2 \times 10^{-6} \text{ mbar}$ before the installation process. The in-situ deposition temperatures and $O_2$ partial pressures in the vacuum chamber were altered leading to the formation of thin films with different crystal structures. The $V_2O_5$ threshold for PLD was enriched with the goodness (99.99 percent) V2O5 powder, with a diameter of 20 mm and a thickness of 2 mm. The UV laser beam cantered by the lens scans the target surface through the quartz window. The angle formed by the incident laser beam and the normal of the target surface was 45 degrees. The laser pulse had an energy of 0.3 Joule and a pulse repetition rate of 10 Hz. After focusing on the target surface, the laser produced a 10 Jcm$^{-2}$. To avoid material depletion at the identical promotion and get identical thin films, the target was interchanged endlessly at a rate of 10 cycles per min. during ablation. The deposition was carried out at a rate of 0.1 nm sec$^{-1}$ on fused silica substrate materials. A thermocouple and temperature controller were used to heat and maintain the substrates in a temperature range of ambient to 300°C. The objective to substrate length was 4 cm, for reactive testimony. During the deposition, purely oxygen gas was released into the chamber through a flow controller. The oxygen limited density was preserved during depositions at 10-2 mbar, which is the optimal assessment to get stoichiometric $V_2O_5$ thin films with a clean phase. All of the PLD $V_2O_5$ thin films used in this study had a thickness of 380 nm. The deposition times were adjusted according to the substrate's nature and temperature to maintain this thickness. Thin films of $V_2O_5$ grown on highly cleaned fused silica substrates were used to investigate the surface morphology and regional composition development as a function of reaction temperature. Diffraction patterns were obtained using the Cu K$_\alpha$ radiation ($\lambda = 1.542\text{A}°$) was employed as the excitation source. The optical transmittance measurements were carried out using a Shimadzu 3101 PC double-beam spectrophotometer in the 320–3200 nm wavelength range. The refractive index (n) was considered using the generalized wavelength of Swanepoel’s [28-30]. The surface morphology of films was observed by scanning electron microscopy (SEM). To determine the oxidation state of the vanadium, in $V_2O_5$ films, X-ray photoelectron spectroscopy (XPS) has been carried out in an analytical system which was activated with an Mg terminal at 10 kV and 10 mA. Before gathering XPS data, no ion bombard has been used to protect any preferred sputtering of the surface organisms. The incidental C1s peak at 284.6 eV was used to correct for any alleging of the sampling surface.

3. Results and discussion

3.1. Optical properties

The values of photosensitive factors [refractive index (n), extinction constant (k), and optical bandgap] as a function of substrate temperatures are summarized in Table 1. It is clear that at higher substrate temperature, the higher values of refractive index and extinction coefficient with lower values of the optical bandgap. In the present work, the values of the optical constants obtained are in respectable settlement with informed values for $V_2O_5$ thin films gotten by several deposition approaches [6-8, 10-11, 16-18].
Table 1. Estimated optical parameter of grown V₂O₅ thin films at several substrate temperatures.

| Substrate temperature (°C) | Refractive index (n) | Extinction coefficient (k) | Optical band gap (eV) |
|---------------------------|----------------------|---------------------------|----------------------|
| Ambient                   | 2.28                 | 0.015                     | 2.36                 |
| 100                       | 2.30                 | 0.021                     | 2.18                 |
| 200                       | 2.49                 | 0.032                     | 2.16                 |
| 300                       | 2.69                 | 0.034                     | 2.08                 |

3.2. Structural properties

By using scanning electron microscopy (SEM) the surface morphology of PLD V₂O₅ thin films was calculated. Figure 1 displays the SEM images of V₂O₅ films as a purpose of growing temperature. The SEM data for V₂O₅ thin films deposited at 300°C show nano-structured grain growth. The film is composed of spherical particles of varying sizes, with an average grain size of 50 nm (Fig. 1A). With increasing temperature, the grain size increased even more. The grains for films deposited at 200°C were almost spherical in shape, but as the temperature rises to 300°C, they definitely moved to a rectangle (Fig. 1B). The rise in grain dimension that is linked to increasing temperature suggests that the grains on the film surface are distributed randomly.

Figure 1. The SEM images of PLD V₂O₅ films as a task of rising temperature: (A) film grownup at 200°C (B) film grownup at 300°C.

Figure 2 shows the relationship between average grain size and evolution temperature. The greater growing temperature leads to the greater grain dimensions. The films dumped at ambient substrate temperature are totally formless. The grain dimension increased from 89nm to 310nm with the rise in substrate temperature from 200°C to 300°C. This could be explained on the basis that, the growth temperature enhanced the surface dispersal of the classes due to the smaller grains connection together and produce into superior grains.
The changing of the bandgap with grain magnitude due to the variation in substrate temperatures is shown in figure 3. It is very clearly show that the upper value of grain size $V_2O_5$ films exhibit a lower value of bandgap. The bandgap value shows a decrease from 2.36 eV to 2.02 eV when the grain size increases from 45nm to 310nm.

The XRD patterns for samples deposited at vary substrate temperatures shows in Figure 4. The crystalline phase of vanadium oxide structure forms at 200°C, as shown in Fig. 2, but only low-intensity peaks can be seen. It is indicated that the crystallinity of the film rises with higher deposition temperatures. The XRD spectrum of the deposited films at 300°C substrate temperature shows peaks that coincided with $V_2O_5$ (001), (400), and (200) exhibit the predominant (001) peak of the orthorhombic $V_2O_5$ phase.
3.3. Compositional Analysis

The binding energy values of the main XPS peaks for V$_2$O$_5$ film samples deposited at 500°C, respectively, are presented in Table 2. As it is seen, V$_2$P$_{3/2}$ and V$_2$P$_{1/2}$ peaks for both samples are similar. The binding energy corresponds to the V$^{5+}$ state of vanadium and agrees well with those reported in[31]. The O1s peak at a binding energy of 530.3 eV corresponding to O$^{2-}$ ions in V$_2$O$_5$ films [31]. In Table 2, the location and spin-orbit split steady for various vanadium oxides are compared to other recorded XPS results. When the experimental data is compared to published data, it is clear that the oxide produced under these conditions is not V$_2$O$_3$, VO$_2$, or any other vanadium oxide other than V$_2$O$_5$[32, 33].

Table 2. Core levels binding energies between levels V$_2$P$_{3/2}$ and V$_2$P$_{1/2}$

| Core level | Experimental data [binding energies (eV)] | Reported data [binding energies(eV)] |
|------------|------------------------------------------|--------------------------------------|
|            | V$_2$O$_5$                               | VO$_2$                               | V$_2$O$_3$                           |
| V$_2$P$_{3/2}$ | 517.1     | 516.9     | 517.6     | 516.1     | 561.2     | 515.5     | 515.7     |
| V$_2$P$_{1/2}$ | 524.2     | 524.5     | 524.3     | 524.4     | 523.1     | 523.5     | 523.0     | 523.3     |

4. Conclusion

In this research, V$_2$O$_5$ films were grown up on a heated fused silica substrate using the PLD method. The effect of substrate temperature was dominant on the film properties. The SEM images of V$_2$O$_5$ films showed the grain dimension is uniform and with mean grain size range from 45nm to 310nm. XPS measurements on the surface designate that the models are mostly collected of V$_2$O$_5$. All the models displayed narrow V$_2$P$_{3/2}$ crests (FWHM~1.6 eV), centred between 517.1 eV and 517.4 eV. The optical band gap for film growth decreased from 2.36 eV to 2.08 eV however, the refractive index increased from 2.28 to 2.69 as the substrate temperature increased from ambient temperature to 300°C.
The grain size showed strong dependence in substrate temperatures. The XRD spectrum of the deposited films at 300°C substrate temperature shows peaks that coincided with V$_2$O$_5$ (001), (400), and (200) exhibit the predominant (001) peak of the orthorhombic V$_2$O$_5$ phase.

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