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Effect of Substrate Temperature on Structural, Optical and Surface Morphological Properties of Spray Deposited V₂O₅Thin Film

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ABSTRACT

Vanadium pentoxide (V_2O_5) thin films have been prepared by spray pyrolysis technique on glass substrate. The films were prepared at 200°C, 225°C, and 250°C. X-ray diffraction analysis revealed orthorhombic crystalline structures of all the film with a preferential orientation along (0 0 1) plane. The effect of substrate temperature on crystallite size and band gap were analyzed and reported. **Keywords:** spray pyrolysis, thin film, V_2O_5 , microstructural



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INTRODUCTION

In recent years vanadium oxide thin films have received significant attention due to their interesting optical, structural and chemical properties [1]. The V- O system also includes a series of oxides with magneli (V_nO_{2n-1}) and wadsley (V_nO_{5n-2})[2] phases. Vanadium oxides obtained from these valences are followed by VO, VO₂, V₂O₃, and V₂O₅. Among these phases, V₂O₅ [1] and has wide applications such as catalyst, gas sensors, electrochromic, thermoelectric devices [3], electronic and optical switches [4]. V₂O₅ thin films have been prepared using various techniques such as electron beam evaporation [5], Magnetron sputtering [6] pulsed laser deposition [7] spray pyrolysis [8], Electrospining and sol –gel method [9]. In the present work spray pyrolysis method has been adopted. This technique offers a list of advantages than other methods such as simple equipment, yields oxide thin films at low cost with high quality andcan deposit effectively large area [10].

Experimental

Vanadium oxide thin films have been deposited onto glass substrate by spray pyrolysis system. A home built spray system was constructed as reported byJeyaprakash et al [11]. A 50ml of 0.05M aqueous solution of Vanadium (III) chloride [VCl₃] was sprayed as fine mist. The solution was sprayed at an angle of 45° onto preheated glass substrate kept at a distance of 50cm from the spray gun. Prior to deposition, the substrate were ultrasonically cleaned. Compressed dry air at a pressure of 2 kg/cm² from an air compressor via an air filter-cum regulator was used as the carrier gas and spray rate of the solution was maintained at 3 ml/min.

To avoid excessive cooling of substrates, successive spraying process was used with time period of 15 seconds between two sprays. Substrate temperature was controlled by chrome-nickel thermocouple fed to a temperature controller with an accuracy of $\pm 1^{\circ}$ C. The temperature on the top side of substrate is measured by placing a thermocouple on a reference glass substrate kept nearer to the coating substrate so as to measure the exact temperature. The structural details of the prepared thin films were carried out. PAnalytical X-ray diffractometer (Model D/MAX ULTIMA III) using Ni- filtered CuK α X- radiation (λ =1.54056Å). A range of 2 θ from 10° to 100° was scanned from a fixed slit type, so that all possible diffraction peaks could be detected. X–Ray line broadening technique was adopted to determine the crystallite size and micro strain. Surface morphology and energy dispersive x- ray spectroscopy (EDAX) of the films was investigated by using FEI Netharland Scanning Electron Microscope (Model QUANTA 200) with an accelerating potential of 18KV. Prior to imaging, the films were sputtered with thin gold film to enhance the emission of secondary electron for better imaging. The optical properties and band gap of the thin films were analyzed by UV-VIS-NIR spectrophotometer (Model-Lambda 35).



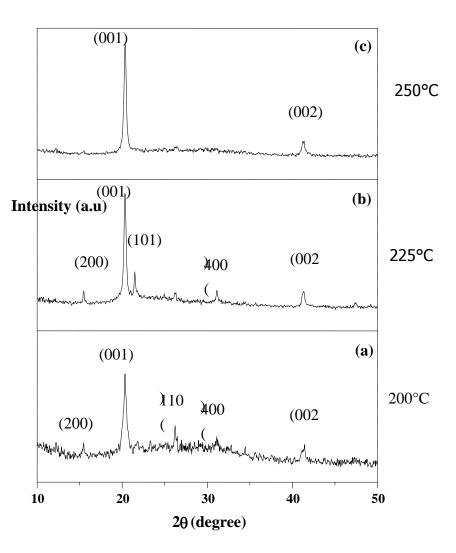


Figure 1 : XRD patterns of V_2O_5 thin films

RESULTS AND DISCUSSION

Structural characterization

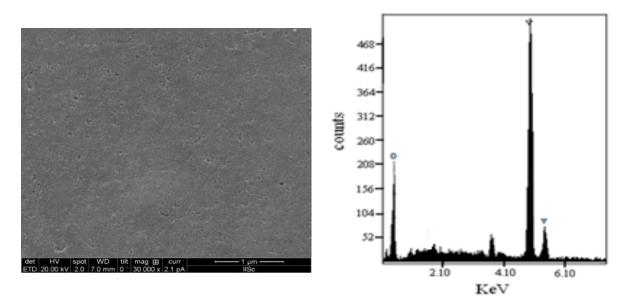
Figure 1 shows the X-ray diffraction (XRD) patterns of prepared V_2O_5 films at various temperatures from 0.05M solution concentration. The obtained XRD patterns aggress with standard JCPDS card [41 – 1426] indicating polycrystalline nature with orthorhombic crystal structure [8]. The random growth or polycrystalline of film is due to amorphous nature of the substrate .The planes were indexed as (0 0 1), (1 1 0), (0 0 2) with respect to standard card.XRD peaks in all the pattern shows a broadening when compared with standard peaks and is due to instrument and specimen microstructural effect. Imperfect optical effects of diffractometer and the wavelength distribution of radiation lead to the instrument



broadening effect and is corrected using standard defect free silicon sample. Whereas the specimen broadening effect is due to grain size and strain. In thin films, strains originate mainly due to lattice mismatch between polycrystalline grains and amorphous substrate and or difference in coefficients of thermal expansion of the film and the substrate. The following well known Scherrer's formula [12] was utilized to determine crystallite size and microstrain.

 $D = \frac{0.9\lambda}{\beta\cos\theta} \text{ and } \qquad \varepsilon_{hkl} = \frac{\beta}{4\tan\theta} \qquad \dots \dots (1)$

WhereD - Size of the grain in the direction perpendicular to the reflectingplanes, θ - Diffraction angle, K - Shape factor (0.9), λ - Wavelength of X-ray, β - The full width at half maximum of prominent peaks in radian and ε_{hkl} -Microstrain. It was observed that as deposition temperature increases crystallite size increases from 20 to 30nm andmicrostrain found to decreases from 8.52 × 10⁻³ to 6.99 × 10⁻³. Figure 2 (a – c) shows the scanning electron micrograph (SEM) and energy dispersive x- ray spectroscopy (EDAX) pattern of V₂O₅ film deposited on glasssubstrate. SEM shows grains with porous structure. Analyzing the EDAX spectrum of V₂O₅ thin film coated on glass substrate at different temperature reveals the presence of vanadium and oxygen in the film. The atomic weight % ratio of (V:O)for the film deposited at 200, 225 and 250°C were 56.35, 43.65at %, respectively. The presence of excess V in the film indicate n- type semiconductor due to electron donor of vanadium metal.





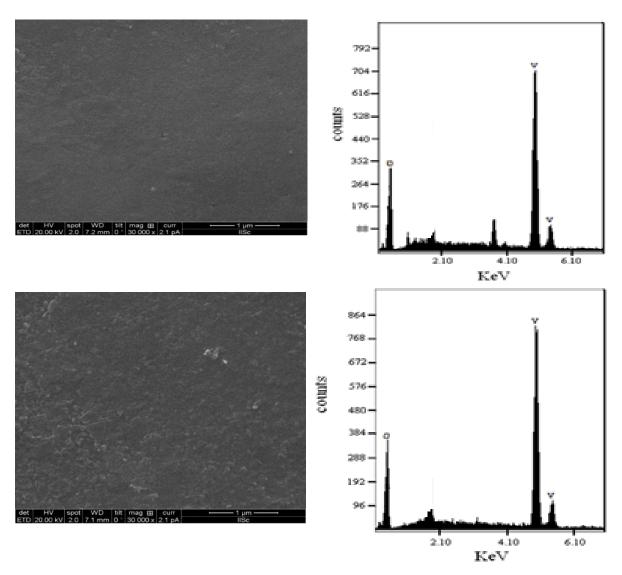
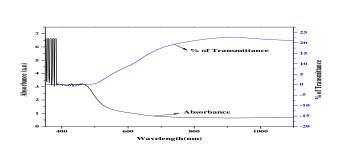


Fig 2 (a-c) SEM and EDAX pattern of V_2O_5 thinfilms obtained at different temperatures

Optical Characterization

Optical absorption measurements were carried out in the wavelength region 350 to 1100nm. Figure 3 (a – c) shows optical absorbance and transmittance of V_2O_5 film prepared at different deposition temperature. All the spectrums show a smooth increase in absorbance above 400nm. This indicates the prepared V_2O_5 film is of high crystalline nature at different temperature.

A graph is plotted between $(\alpha hv)^2$ and hvto determine direct allowed band gapsas shown in figure 4. The straight portion of the graph is extrapolated to energy axis to give E_g value and its value is found to be 2.32, 2.38 and 2.46eVfor different temperature [13].



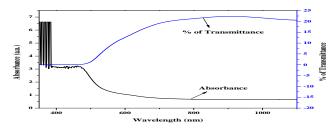


Figure 3 (a-c): UV – VIS Absorbance and Transmittance of V_2O_5 thinfilms obtained at different temperatures

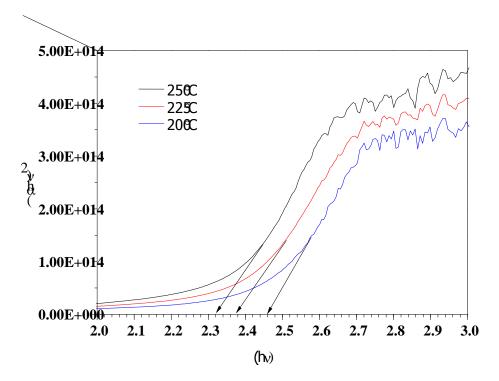


Figure 4: Plot of $hv / (\alpha hv)^2$ of V_2O_5 prepared at different

CONCLUSION

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Vanadium oxide thin films were prepared by home built spray pyrolysis unit on glass substrate at different temperature. It was observed that the film was of polycrystalline in nature with orthorhombic crystal structure and has preferential orientation along (0 0 1)plane. Optical band gap strongly depends on substrate temperature. The presence of porous over the surface of the spray deposited V_2O_5 film indicates good material for chemical sensing application.

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