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Plasma Assisted Synthesis of Vanadium Pentoxide Nanoplates

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Abstract. In this work, we report the growth of α-V_2O_5 (orthorhombic) nanoplates on glass substrate using plasma assisted sublimation process (PASP) and Nickel as catalyst. 100 nm thick film of Ni is deposited over glass substrate by thermal evaporation process. Vanadium oxide nanoplates have been deposited treating vanadium metal foil under high vacuum conditions with oxygen plasma. Vanadium foil is kept at fixed temperature growth of nanoplates of V_2O_5 to take place. Samples grown have been studied using XPS, XRD and HRTEM to confirm the growth of α-phase of V_2O_5, which revealed pure single crystal of α- V_2O_5 in orthorhombic crystallographic plane. Surface morphological studies using SEM and TEM show nanostructured thin film in form of plates. Uniform, vertically aligned randomly oriented nanoplates of V_2O_5 have been deposited.

INTRODUCTION

Low dimensional transition metal oxide have gained great interest due to their unique physical, electronic, chemical properties. Vanadium pentoxide (V_2O_5), one of the semiconductor material, has attracted attention during the last decades primarily, owing to its fascinating applications in many field, such as, electrochromism, gas sensing, power storage devices (Li-ion battery), field emission devices and catalysis. Multivalency, layered structure, good chemical and thermal stabilities are the characters make a promising material in various applications mentioned above. However there are challenges in growth of nanostructured material, such as high surface to volume ratio, structural stability etc. which needs proper attention in order to use material for applications. Till now, various synthesis techniques have been explored for V_2O_5, such as sol-gel method [2], thermal evaporation method [2], hydrothermal method [1] and electrochemical deposition method [2]. Based on these method, various kind of nanostructures have been obtained. Most of the research groups, used chemical route with or without post treatments but very limited reports have published on synthesis of V_2O_5 nanostructured thin film using PVD approach. In this report, we have achieved the growth of nanoplates by a relatively new PVD route named plasma assisted sublimation process [4-5].

EXPERIMENTAL

First, nearly 100 nm thick Ni-film is deposited on glass substrate using thermal evaporation process of 99.99% pure Ni powder at base vacuum of 7.5×10^{-6} Torr. This is succeeded by the deposition of vanadium oxide nanostructured thin films. The experimental setup of plasma assisted sublimation route have two cup-shaped Al–electrodes facing each other and placed at a separation of 75 mm. For generation and control of plasma, current is supplied by power supply. Vanadium metal foil is used as source material for deposition process. Nickel coated glass substrate is place over metal foil inside the vacuum chamber as shown in Fig. 1. The vacuum chamber is evacuated to 7.5×10^{-6} Torr. Afterwards, high purity oxygen gas is inserted into the chamber and oxygen partial pressure is kept at optimized value. The flow of oxygen is maintained to sustain stable plasma. All the plasma parameters are kept constant at the time of film deposition. Vanadium metal foil is heated up to a temperature of

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450 ºC. During the growth process, vanadium reacts with oxygen species present in the chamber to form V_2O_5, which gets deposited on substrate. The growth process continues for 40 min.

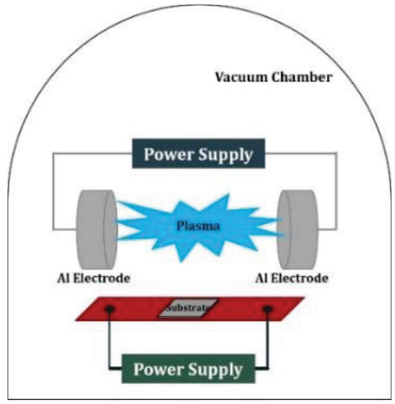


FIGURE 1: Schematic representation of plasma assisted sublimation process

The surface microstructure of all the deposited nanostructured films is studied with scanning electron microscope ZEISS model EVO-50. Transmission Electron Microscopy (TEM) study of V_2O_5 nanoplates is carried out with Philips Model CM12 operating at 120kV with HRTEM analysis. Structural studies are carried out using Philips X-ray diffractometer with Cu-K_α radiation (λ~1.54Å) source and glancing angle constant at 0.5º. Photoelectron spectroscopic studies is carried out by SPECS, with anode Mg/Al 25kV X-ray source and a hemispherical analyzer PHOIBOS HSA3500 150 R6 [HW Type 30:14] MCD-9. All the measurements are performed at room temperature.

RESULTS AND DISCUSSION

In order to study the crystal composition phase purity of vanadium pentoxide nanostructure thin film, x-ray diffraction pattern of samples have been recorded is shown in figure 2.

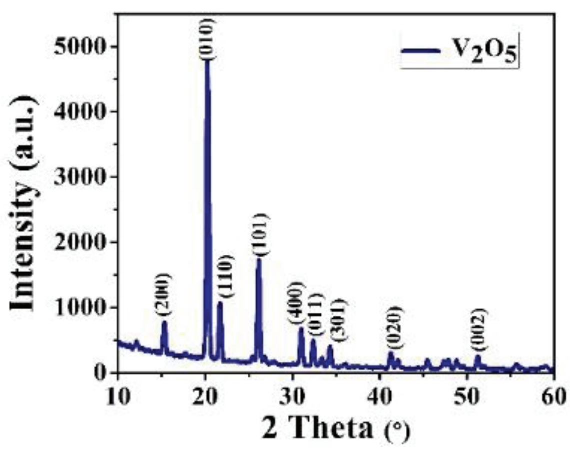
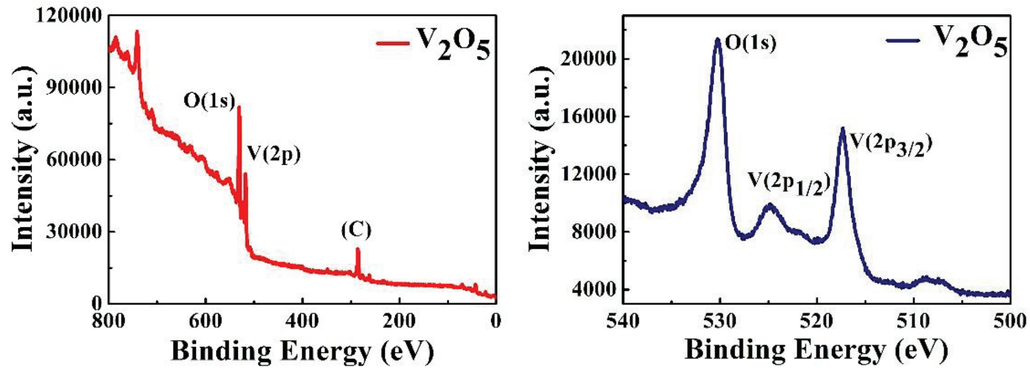


FIGURE 2: X-Ray Diffraction pattern of V_2O_5 nanoplates grown by plasma assisted sublimation process

It is observed, that XRD pattern have sharp peaks showing polycrystalline nature of film. The planes have been identified by JCPDS (ref code: 86-2248). The study of observed highly intense diffraction peak in all samples shows the presence of orthorhombic phase. It is to be noted that no impurity phase was found under the resolution of XRD. XRD pattern revealed highly preferred growth oriented along [010] crystallographic direction. The lattice parameter calculated from XRD analysis are a = 3.56 Å, b= 11.51 Å and c = 4.37 Å.

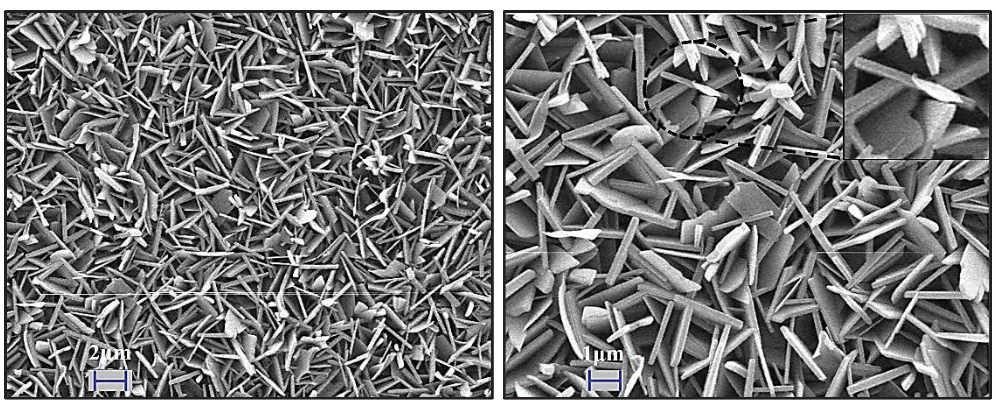
In order to study the material quality and chemical compositions XPS analysis have been carried out. Figures 3(a) shows the general XPS survey scan, in which three intense peaks corresponding to oxygen (1s), vanadium (2p) and carbon (1s) (which is calibrated to carbon peak position at 284.6 eV) have been found. These peaks are in accordance to the reported values to the core level binding energies. In the survey scan, no element other than vanadium, oxygen and carbon was found, which confirms purity of vanadium oxide nanoplates. Core level scan of vanadium-oxygen is shown in Fig. 3(b).



(a) (b)

FIGURE 3 : (a) Survey scan of V_2O_5 nanoplates. (b) core level scan on vanadium pentoxide showing 5^+ oxidation state confirming V_2O_5.

Here, oxygen 1s peak is observed at 530 eV. The splitted vanadium 2p level at binding energy 517.2 eV and 524.8 eV are representing to doublet states of vanadium 5^+, $2p_{3/2}$ and $2p_{1/2}$ respectively. The binding energy separation is 7.5 eV [3], which is in agreement with results reported earlier. The vanadium peak 517.0 eV corresponds to 5^+ oxidation state, so the presence of V_2O_5 is inferred, which is confirmed by results of XRD.



(a) (b)

FIGURE 4: SEM micrograph of nanoplates of vanadium pentoxide films at (a) 5,000x and (b) 10,000x magnification. Micrographs shows uniform growth of nanoplates over substrate.

Figures 4(a) and 4(b) show micrograph of uniform V_2O_5 nanostructured thin film deposited on glass substrate at 5,000x and 10,000x magnification respectively. The nanostructures are plate like as seen in figures. These nanoplates distributed uniformly over the substrate. The density of nanoplates doesn't vary over the surface under observation. Nanoplates of V_2O_5 are vertically aligned over the substrate, but have random orientation. It is also observed that nanoplates have a narrow size distribution.

V_2O_5 nanoplates are further characterized for detailed study of structure and crystallinity. TEM micrograph is shown in Fig. 5. It can be observed that the smooth edges of nanoplates with thickness of about 90 nm. High resolution image (inset of Fig. 5) of V_2O_5 shows fringe pattern indicating single crystalline nature of nanoplates. The spacing between fringes are measured and value nearly matches with d_{010} interplanar spacing. This further indicates the growth of nanoplates is preferred along b-direction.

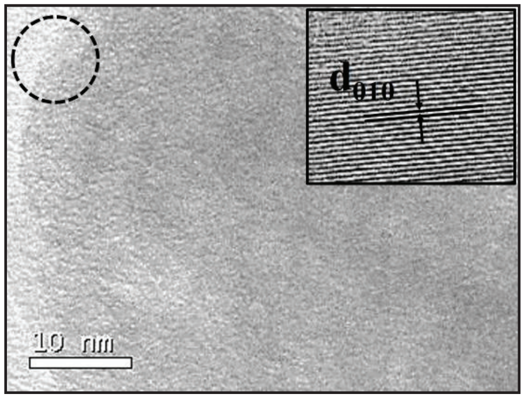


FIGURE 5: TEM micrograph; inset: High resolution TEM image of V_2O_5 nanoplates.

CONCLUSIONS

To summarize, nanoplates of α-V_2O_5 (orthorhombic) have been deposited on Ni coated glass substrate using plasma assisted sublimation process. The structural studies on samples confirm the growth of vanadium pentoxide with oxidation state 5^+ indicating stoichiometric growth. The nanoplates are vertically aligned, randomly oriented with uniform size distribution over substrate. Further detailed study indicated the crystalline nature of nanoplates with preferred growth direction along [010] plane. The results of HRTEM, XRD and XPS corroborates with results reported earlier.

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