# Growth of α-V2O5 nanostructured thin films as a function of deposition process

Megha Singh, Rabindar K. Sharma, and G. B. Reddy

Citation: AIP Conference Proceedings **1731**, 050041 (2016); doi: 10.1063/1.4947695
View online: http://dx.doi.org/10.1063/1.4947695
View Table of Contents: http://scitation.aip.org/content/aip/proceeding/aipcp/1731?ver=pdfcov
Published by the AIP Publishing

# Growth of α-$V_2O_5$ Nanostructured Thin films as a Function of DepositionProcess

Megha Singh*, Rabindar K. Sharma, and G. B. Reddy

*Thin film Laboratory, Department of Physics, Indian Institute of Technology Delhi -110016*
*Email: *meghasingh.life@gmail.com*

**Abstract.**In this communication, we synthesizedvanadium pentoxide (α-$V_2O_5$) nanostructured thin films ($NST_s$) using four different methods for obtaining vanadate species namely thermal evaporation (source of vanadate species are $V_2O_5$ powder and vanadium metal foil) and plasma assisted sublimation process (source of vanadate species are $V_2O_5$ powder and vanadium metal foil). The effect of plasmaon morphological and structural propertieshave been systematicallystudied. XRD revealed thermal evaporation process yielded amorphous films whereassublimation process yielded highly crystalline α-$V_2O_5$ films. HRTEM of nanobelts show, the growth is preferred in (001) crystallographic direction with interplanar distance of 0.43 nm. XPS revealed O/V ratio of ~2.4, which nearly agrees with standard $V_2O_5$ stoichiometry. SEM revealed deposition process affect morphology of films; thermal evaporation results in smoother film while plasma assisted sublimation process reveals nanoflakes and nanobelts (NBs).All the results are inconcordance with each other.

**Keywords:** Vanadium pentoxide, Plasma Voltage, Plasma assisted sublimation process
**PACS:** 68.37.Og, 68.55.-a, 81.07.-b

## INTRODUCTION

Vanadium pentoxide ($V_2O_5$) especially in the form nanostructured thin films (NSTs) has attracted attention of researchers primarily owing to its fascinating application in various kinds of applications like in gas sensing, as a window in solar cell, electrochromic devices, power storage devices (Li+-ion batteries), catalysis, as well as in electronic and optical switches due to their structural, electronic, optical and electrical properties. Wide optical band gap and good thermal and chemical stabilities are the characteristics, making $V_2O_5$ as a promising material in various applications mentioned above. There are several approaches, used to grow various kind of vanadium oxide nanostructures such as thermal evaporation, pulse laser deposition, spray pyrolysis, etc. Beke *et al*. have reported the nanoclusters of $V_2O_5$ on glass substrate using pulsed laser deposition and studied their structural and optical properties.[1] Chen *et al.* deposited $V_2O_5$ thin film at different temperature by sol gel technique.[2] C. Diaz-Guerra *et al*. have reported the growth of $V_2O_5$ nanowires and nanotips on Si substrate.[3] Herein, we report the synthesis of $V_2O_5$ NSTs by physical vapour deposition technique (PVD) and studied the intrinsic parameters that can be controlled during film deposition, such as source of vanadium oxide, and the presence of oxygen plasma in the evolution of vanadate species. The films grown using two different deposition processes are characterized and analyzed.

## EXPERIMENTAL SETUP

In order to study the effect of vanadate source on the deposited nanostructured thin films, samples were prepared in four different configurations were employed summarized under labels M1-M4. The experimental setup is essentially thermal evaporation system modified to incorporate assembly for plasma generation and stabilization.Prior to deposition glass substrates were coated with Ni, to act as catalyst in the growth of nanostructures. The growth temperature for all experiments is constant at 450 ºC. All plasma parameters, plasma voltage (2500 V), separation between electrodes (70 mm), gas flow rate (where plasma was used) were kept constant throughout film deposition. Duration of deposition process is kept 30 minutes for all samples.

Sample preparationM1:$V_2O_5$ powder was thermally evaporated in oxygen environment to deposit thin films over substrate.

Sample preparation M2:Vanadium oxide species were obtained by oxidizing vanadium metal foil (5 cm x 1 cm x 0.5 mm) in presence of oxygen environment prior to deposition. After the oxidation has taken place

*DAE Solid State Physics Symposium 2015*
AIP Conf. Proc. 1731, 050041-1–050041-3; doi: 10.1063/1.4947695
Published by AIP Publishing. 978-0-7354-1378-8/$30.00

glass substrate was kept in vacuum chamber and metal foil was heated to transport these vanadium oxide species onto the substrate.

Sample preparation M3: $V_2O_5$ powder was first made into pellets which wereused as sublimation source for vanadatespecies in oxygen plasma.

Sample preparation M4:Vanadate species were obtained from the surface of oxidized vanadium metal foil. The transport of oxide species were supported by presence of oxygen plasma. This process is known as plasma assisted sublimation process.

ZEISS EVO series scanning electron microscope model EVO-50 was used for studying surface morphology. Transmission Electron Microscopy (TEM) study of $V_2O_5$NST$_s$wasconducted with Philips Model CM12 with HRTEM analysis.Photoelectron spectroscopic studies were carried outby SPECS, with anode Mg/Al 25kVX-ray source.All the measurements are performed at room temperature.Rigaku Ultima IV model X-ray diffractometer with Cu-Kα radiation (λ ~ 1.54Å) source with glancing angle constant at 1° employed for structural analysis.

## RESULTS AND DISCUSSION

X-ray diffractograms (XRD) are recorded to investigate the composition and crystallininityphase of vanadium oxide thin films deposited through different deposition processes, as shown in Fig. 1. Sample M1 is purely amorphous in nature as no peaks were observed inX-ray diffractogram.Whereas, diffractogram of sample M2, clearly depicts amorphous films with slight degree of crystallinity as XRD pattern have a large hump in the 2θ range of 20° to 30° along with small peaks at 2θ values 15.33° and 26.09°.

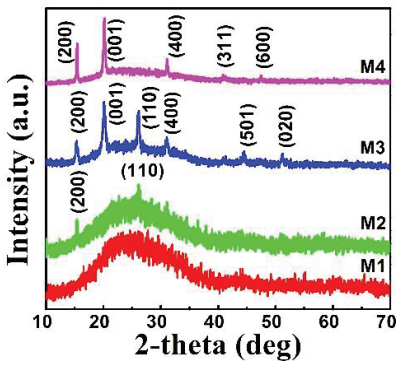


**FIGURE 1**. X-ray Diffraction of samples M1-M4

The sharp diffraction peaks in the diffractograms of samples M3 and M4 are directly demonstrate the polycrystalline nature with orthorhombic phase of vanadium pentoxide films.Any phase other than α-$V_2O_5$were not found under the resolution limit of XRD. The obtained values of lattice parameters from XRD analysis are a = 11.51Å, b = 3.56Å and c = 4.37Å, which are quite in good agreement with the reported values as given in JCPDS (Ref. Code: 89-0612) a = 11.544 Å, b = 3.571 Å and c = 4.383 Å for α-$V_2O_5$. Films deposited by thermal evaporation (M1 and M2) are amorphous because impinging flux sticking to the substrate surface at its place of hitting with almost no surface diffusion and hence the formation of amorphous $V_2O_5$ films. But in case of sample M3 and M4, plasma played an important role to transfer oxide species. Since, it has been reported earlier that the presence of $O_2$plasma not only facilitates in oxidation of (V-metal strip for M4) at relatively lower temperature but also directing the $V_2O_5$ molecules to reach on to substrates and distribute these over the large area of substrate (1.5 cm x 1.5 cm) in a homogenous manner.[4] Therefore, in presence of plasma the vandate species acquire higher energy and hence a large mobility. As a result, the diffusion distance of the adatom on the surface increases and the collision process initiates the nucleation for more adatoms joining to form larger grains.Figure 2 depicts the surface morphology of samples M1, M2, M3, and M4. The SEM micrograph of M1 exhibits smooth surface. Also, micrograph of M2 shows smooth surface with sparse flake-like features.

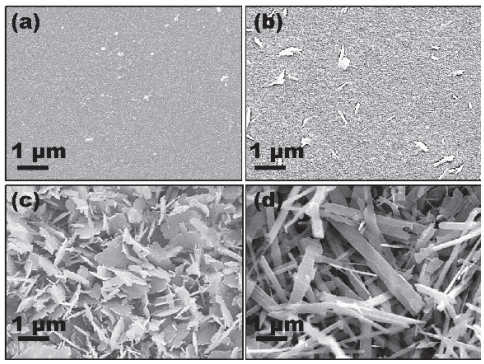


**FIGURE 2**. SEM images $V_2O_5$ nanostructured thin films (a) M1, (b) M2, (c) M3 and (d) M4

In case of M3,source $V_2O_5$ powder sublimes in plasma environment furnishes enough energetic moleculesof$V_2O_5$ for throughout deposition. For sublimation carried out in presence of plasma,using V-metal foil, nanobelt (NBs) structures are obtained. The well-established feature in case of sample M4 mainly because of better rate of oxidation as well as high energy of the volatilized $V_2O_5$ species from vanadium-metal foil during deposition, which leadsto form the well aligned nanostructures. The average length of

nanobelts is in the order of few microns. All these NBs are uniform along with the average width of the order of 100 nm.It is speculated the existence of the oxygen vacancies at top edges of NBs grown in oxygen plasma offered the nucleation site for the further incoming $V_2O_5$ molecules to condense. This will enable the belts to grow rapidly along its length compare to the other directions. Therefore, it is concluded that proper energy of the incoming oxide is necessary to obtain better features and alignment of NSTs.In further analysis, we selected the best sample (which is M4 in this case) in order to obtain more insight information of grown nanostructure through TEM in bright field mode accompanied with HRTEM, as shown in Fig.3 Here TEM image of NBs confirms that nanobelts have well defined dimensions, which is nearly matched with SEM observations from dimensional prospective. In addition, the fringe pattern(high resolution image) is recorded by HRTEM from the encircled region marked on NBs that corroborated the crystalline nature of NBs, indicates that the lattice spacing is about 0.43 nm. This distance is consistent with the (001) plane of the orthorhombic structure of $V_2O_5$, as viewed in the inset of Fig. 3.

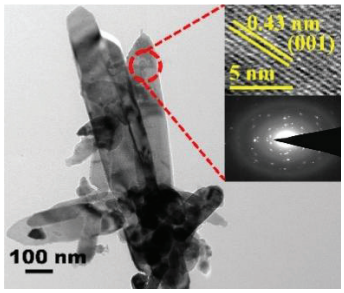


**FIGURE 3.** Bright field TEM micrographs of $V_2O_5$ NBs

The XPS survey spectrum is acquired from the surface of $V_2O_5$ NBs (sample M4), revealed only three intense bands of V (2p), O (1s), and C (1s).

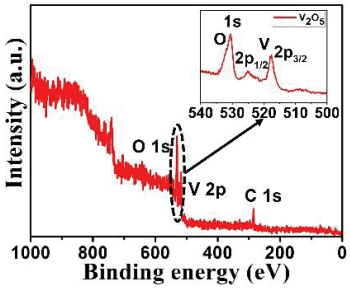


**FIGURE 4.** XPS of $V_2O_5$ nanobelts deposited at growth temperature (450 ºC)

The binding energies of doublet V 2p are 516.8 eV and 524.4 eV for V $2p_{3/2}$ and V $2p_{1/2}$ respectively, with a splitting of 7.6 eV and O1s level is present at 529.8 eV. Calibration is done by Carbon 1s peak at 284.6 eV. The absence of any other elemental peaks, confirming the purity of the $V_2O_5$thin film. To get more information core level scan of V (2p) and O (1s) is carried out, as seen in inset of Fig 4. The calculated ratio of the O/V is ~2.4, which is nearly in good agreement with the standard stoichiometry ratio in $V_2O_5$.

## CONCLUSIONS

To summarize, $V_2O_5$ films deposited on Ni coated glass substrates using thermal evaporation in oxygen using $V_2O_5$ powder (M1), using V-metal foil (M2), sublimation in oxygen plasma using $V_2O_5$ powder (M3) and sublimation using V-metal foil (M4). The SEM micrographs clearly show the effect of deposition process on surface morphology of films. Samples deposited using thermal evaporation show near smooth surface, whereas, samples M3 and M4 exhibit nanoflakes and nanobelts, respectively. XRD results also show the effect of deposition process on structural properties of film. Thermal evaporation process in oxygen ambient resulted in amorphous $V_2O_5$ films, whereas plasma assisted sublimation process resulted in crystalline films. The compositional results show highly pristine $V_2O_5$ films with O/V ratio of ~ 2.4, which nearly agrees with standard stoichiometry ratio. High resolution image obtained from HRTEM of $V_2O_5$ nanobelts show an interplanar distance of 0.43 nm, confirming growth along (001) direction which is also the plane with most intense peak in XRD. Hence, it is concluded that deposition process greatly affect crystallinity and morphology of $V_2O_5$ thin films.

## ACKNOWLEDGMENTS

We acknowledge the Nanoscale Research Facility, and use of XPS facility (partially funded by FIST grant of DST) at I.I.T. Delhi.

## REFERENCES

1. S. Beke, S. Giorgio, L. Kőrösi, L. Nánai, W. Marine, Thin Solid Films **516**, 4659–4664 (2008).
2. Yu Chen, Cheng Chen, Wei Chen, Heng Liu, Jianguo Zhu, Progress in Natural Science: Materials International **25**, 42-46 (2015)
3. C. Díaz-Guerraa and J. Piqueras, Journal of Applied Physics **102**, 084307 (2007)
4. Rabindar K. Sharma, Prabhat Kumar, G.B. Reddy, J. Alloys Comp **638,** 289–297 (2015)