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A Comparative Study: Effect of Plasma on V_2O_5 Nanostructured Thin Films

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Abstract: Vanadium pentoxide nanostructured thin films (NSTs) have been studied to analyze the effect of plasma on nanostructures grown and morphology of films deposited using sublimation process. Nanostructured thin films were deposited on glass substrates, one in presence of oxygen plasma and other in oxygen environment (absence of plasma). Films were characterized using XRD, Raman spectroscopy, SEM and HRTEM. XRD studies revealed α-V_2O_5 films (orthorhombic phase) with good crystallinity. However, film deposited in presence of plasma have higher peak intensities as compared to those deposited in absence of plasma. Raman studies also support these finding following same trends of considerable increase in intensity in case of film deposited in presence of plasma. SEM micrographs makes the difference more visible, as film deposited in plasma have well defined plate like structures whereas other film have not-clearly-defined petal-like structures. HRTEM results show orthorhombic phase with 0.39 nm interplanar spacing, as reported by XRD. Results are hereby in good agreement with each other.

INTRODUCTION

Ever since the inception of manipulation of atoms, a new class of materials called nanostructures have come center stage. Material scientists have been studying the properties of these nanostructures, in order to control and modify them for newer applications. Of this class, a separate category of transition metal oxides have become popular, primarily due to their varying oxidation states leading to interesting properties with fascinating applications. Vanadium pentoxide is a layered structured material with promising applications in catalysis, electrochromism, electrochemistry [1,2], cathode material lithium-ion batteries [3], etc. V_2O_5 show metal-insulator transition above room temperature, opening up new frontiers for applications in semiconductor devices. All these applications make V_2O_5 an interesting material for to be studied, particularly nanostructured V_2O_5 films. Here, we present a novel method of synthesis of V_2O_5 nanostructures namely, plasma assisted sublimation process (PASP). Using this method, nanostructured thin films (NSTs) of V_2O_5 have been prepared in oxygen plasma. Present report focusses on effect of presence of plasma during the growth of V_2O_5 NSTs.

EXPERIMENTAL

Experimental setup for plasma assisted sublimation process (PASP) consists of heating assembly for sublimation source of vanadium oxide and plasma assembly for generation and control of plasma. For sublimating source, tungsten metal foil is provided controlled current via external 10 A power supply. V_2O_5 pellet (acting as vanadium oxide source) is placed over tungsten foil. Glass substrate is placed (as shown in fig. 1) 5 mm over V_2O_5 pellet such that it lies in the field of plasma.

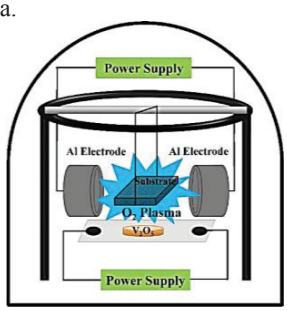

FIGURE 1: Schematic representation of plasma assisted sublimation process

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Plasma assembly consist of aluminium cup-shaped electrodes provided voltage controlled externally by a separate power supply. Pressure is maintained at 7.5×10^{-6} Torr. Oxygen gas is inserted in chamber and partial pressure is maintained at 1.2×10^{-1} Torr. Plasma voltage (2500 V) is applied to electrodes for generation of O_2 plasma. Source is heating up to temperature 350 ºC. Growth process continues for 30 minutes. During growth process, V_2O_5 powder sublimes in oxygen plasma, thereby maintaining the oxygen/ vanadium ratio in final deposited NSTs. The role of oxygen plasma on oxidation of vanadium metal (foil) and volatilization of oxides have already been studied by Rabindar K. Sharma *et. al.*[4] In order to study the effect of plasma on growth of V_2O_5 NSTs, samples were deposited in presence of O_2 plasma (S2) and in absence of plasma (S1). Samples thus deposited were characterized.

Surface morphology and microstructure of deposited nanostructured films were studied with scanning electron microscope ZEISS model EVO-50. Transmission electron microscopy (TEM) study of V2O5 nanoplates was carried out with Philips Model CM12 with HRTEM analysis. Structural studies are carried out using Philips X-ray diffractometer with Cu-Kα radiation (λ~1.54Å) source. All the measurements are performed at room temperature. Vibrational studies of synthesized films were carried out by Raman spectroscopy (Horiba Lab RAM HR Evolution) equipped with Ar ion 514 nm laser at 20 mW power.

RESULTS AND DISCUSSION

X-ray diffractograms are recorded to investigate the composition and phase purity of V_2O_5 thin films deposited on glass substrate for 30 min in presence and in absence of plasma. It is observed that, XRD pattern have sharp and intense peaks indicating polycrystalline nature of films. In sample S1 and S2, the diffraction peaks in XRD pattern are indexed according to the pure orthorhombic crystalline phase of V_2O_5, which are corresponding to the standard value as reported in JCPDS file (89-0612) with the lattice constants of a = 11.48 Å, b = 3.55 Å, and c = 4.36 Å and no traces of other characteristics peaks corresponding to impurity phases is detected under x-ray diffractogram.

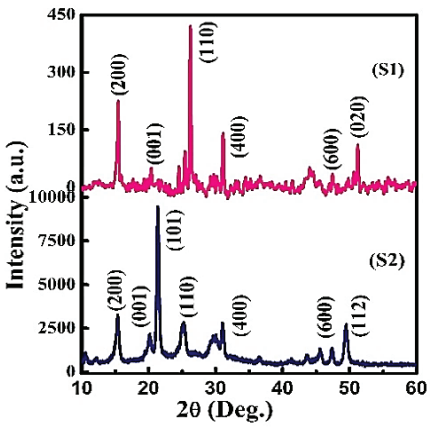

FIGURE 2: X-Ray Diffraction pattern of V_2O_5 films (S1) in O_2 ambience and (S2) in presence of O_2 plasma

The most intense peak are obtained at 2θ value = 26.09º corresponding to (110) crystal plane in sample S1. Whereas in sample S2 the intensity of all the peaks get increased significantly as compared to sample S1. This change is attributed to sufficient gain of energy in plasma ambient by vanadium pentoxide molecules to organize themselves in orderly fashion. And hence forming highly crystalline structure, which is reflected in sharpness and intensity of obtained peaks (as shown in fig. 2).

The results of XRD are further supported by SEM micrographs. The recorded micrograph of film deposited on glass in sample S1 shows the growth of randomly aligned petal-like nanostructures with average length and width of 200 nm and 50 nm, respectively (see in Fig. 3). In contrast, the V_2O_5 film deposited on glass substrate (S2) in presence of plasma shows plate-like nanostructures. The grown nanostructures are more defined and regular with average length and width of 200 nm and 100 nm respectively and are distributed uniformly throughout the sample.

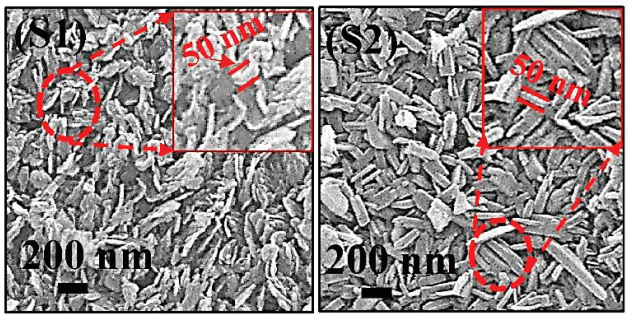

FIGURE 3: Micrographs of films deposited (S1) in O2 ambience and (S2) in presence of O2 plasma.

Raman spectroscopy was carried out to study the vibrational properties of samples S1 and S2 in the spectral range of 100 - 1020 cm^{-1}. Figure 4 shows the details of Raman active peaks of V_2O_5 with the vibrational bands located at 142, 194, 282, 406, 483, 525, 700, and 991 cm^{-1}, which are assigned to modes of polycrystalline orthorhombic phase of V_2O_5.

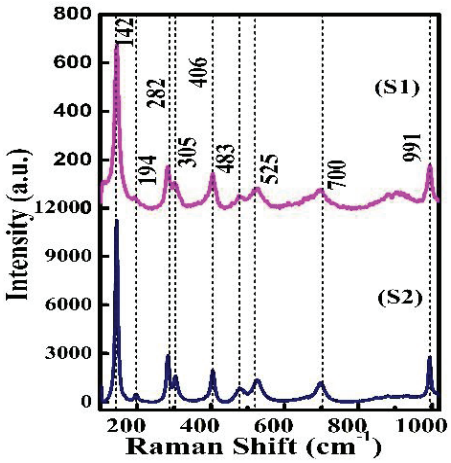

FIGURE 4 : Raman shift of samples S1 and S2.

The most intense peak obtained at 147 cm^{-1} is attributed to the skeleton bent vibration and also evidencing the layered structure of V_2O_5. The peaks present at 282 and 406 cm^{-1} are assigned to the bending vibrations of V=O bonds. The vibrations corresponding to triply coordinated oxygen (V3-O) stretching is observed at 525 cm^{-1} whereas the peak at 700 cm^{-1} is assigned to the doubly coordinated oxygen (V2-O). The peak present at 991 cm^{-1} is due to the terminal oxygen (V=O) stretching mode. In case of sample S2 the peak intensity of the peaks are relatively much higher than in case of sample S1 which suggest the improvement in the crystallinity and supports results obtained from XRD.

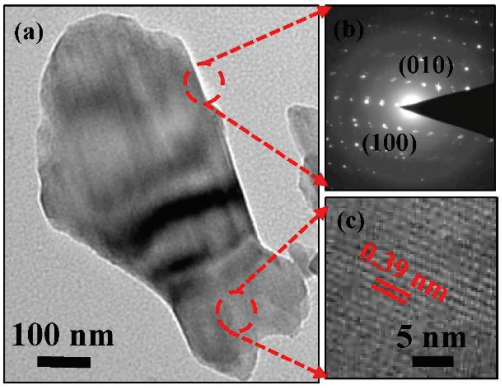

FIGURE 5: (a) TEM micrograph of nanoflake; (b) SAED pattern; (c) High resolution TEM image of V_2O_5 nanoflake.

In sample S2, deposited V_2O_5 nanoplates are further characterized using HRTEM (fig. 5). It is observed that nanoplates obtained in said sample have smooth edges. This observation is quite consistent with that obtained from SEM micrographs. Selected area electron diffraction pattern (see in Fig. 5b) divulges that V_2O_5 nanoplates composed of a single crystalline orthorhombic phase. The interplanar spacing measured (from high resolution image of V_2O_5; Fig. 5c) to be 0.39 nm and value nearly matches with interplanar spacing of plane (101). All the results thus support each other.

CONCLUSION

In summary, V_2O_5 NSTs were deposited on glass substrates; one is presence of plasma and other in absence of plasma. The samples were studied to find out effect of plasma on growth of V_2O_5 NSTs. It was inferred by XRD pattern of S1 and S2 that films deposited in presence of plasma have improved crystallinity which was later confirmed by Raman analysis of the samples. SEM micrographs showed that films deposited in absence of plasma lacked property defined structures as in contrast with film deposited in presence of plasma. HRTEM results supported the results obtained by XRD. Therefore, it is concluded that plasma yields properly defined nanostructure with enhanced crystallinity.

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