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Electrochromic properties of vanadium oxide thin films prepared by PSPT: Effect of substrate temperature

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Abstract. Electrochromic vanadium oxide (V_2O_5) thin films were deposited onto glass and fluorine doped tin oxide (FTO) coated glass substrates from methanolic vanadium chloride solution by pulsed spray pyrolysis technique (PSPT). The films were synthesized at different substrate temperatures ranging from 350°C-450°C with a temperature step of 50°C. The structural, morphological, optical and electrochromic properties of the synthesized films were investigated. The films were polycrystalline with tetragonal crystal structure. Scanning electron microscopy reveals compact morphology at high temperature. All films exhibited cathodic electrochromism in lithium containing electrolyte (0.5 M $LiClO_4$ + Propylene Carbonate). Maximum coloration efficiency (CE) 15.16 cm^2C^{-1}, was observed for the films deposited at 350°C.

Keywords: Vanadium oxide; Pulsed spray pyrolysis technique; X-ray diffraction; Electrochromism;
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INTRODUCTION

Electrochromism (EC) is a phenomenon in which materials are able to change their optical properties in a reversible and persistent way under the action of a voltage pulse. Transition metal oxides have been widely used as inorganic electrochromic materials and are based on a reversible change in the optical properties during electrochemical oxidation or reduction processes [1].

Vanadium oxide (V_2O_5) as an electrochromic counter electrode has been reported [2-3]. V_2O_{5-x} film in a colored state (yellow) can be reversed to a bleached state (light blue) by both insertion of ions and electrons to undergo intervalence charge transfer according to insertion/extraction reaction as [4-5]:

$$V_2O_{5-x} + xM^+ + xe^- \leftrightarrow M_xV_2O_{5-x} \quad (1)$$

where, M^+ denotes H^+, Li^+, Na^+ or K^+ ions.

In this study, we investigated the structural, morphological, optical and electrochromic properties of V_2O_5 thin films deposited by PSPP at various substrate temperatures. PSPT has several advantages such as simplicity, safety, low cost of the apparatus and raw materials as well as large scale deposition.

EXPERIMENTAL

V_2O_5 thin films were deposited by PSPT onto both glass and FTO-coated glass substrates at different substrate temperatures (350, 400 and 450°C) as reported earlier [6]. In brief, the precursor solution was sprayed on to the pre-heated glass substrates maintained at a desired substrate in which 20 ml of methanolic VCl_3 solution was sprayed. The V_2O_5 thin films deposited at 350, 400 and 450°C are denoted by V_{350}, V_{400} and V_{450}, respectively.

The electrochromic properties carried out in three electrode system, in which the, V_2O_5 thin film deposited on to FTO coated glass substrate (15-20 Ω cm^{-1}), a graphite plate and a saturated calomel electrode (SCE) served respectively as working electrode, counter electrode and the reference electrode. A 0.5 M $LiClO_4$ + Propylene Carbonate was used as an electrolyte solution.

RESULT AND DISCUSSION

Fig.1 (a) shows the SEM image of V_{350} sample which reveals the compact morphology of V_2O_5 devoid of any cracks with whitish lump of overgrowth. Fig. 1 (b) shows the SEM image of V_{400} sample which shows the overgrowth which reduces the cracks

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slightly. And for highest substrate temperature of 450°C (sample V_{450}) as shown in Fig.1 (c), the surface becomes quite smooth and compact. Fig. 1 (d) shows the XRD patterns of the V_{350}, V_{400} and V_{450} samples over 20°-80°. The observed XRD patterns are compared with the standard diffraction data (lattice parameters; a=14.259 Å, b=14.259 Å and c=12.576 Å) [29]. Well-resolved peaks characterizing the tetragonal crystal structure are observed in the XRD patterns of V_2O_5 thin films.

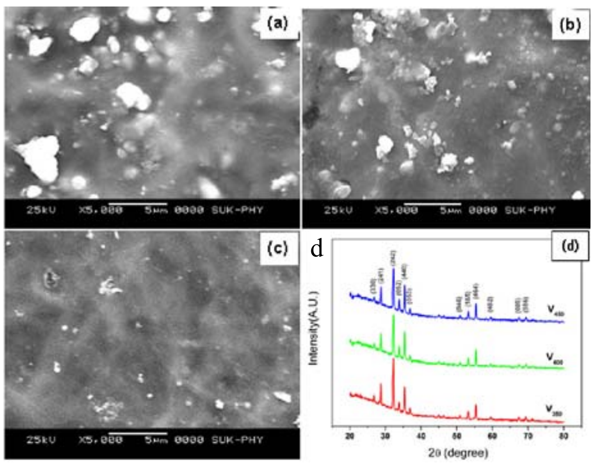

FIGURE 1. SEM images for the (a) V_{350}, (b) V_{400} and (c) V_{450} samples and (d) is overlay of all XRD patterns

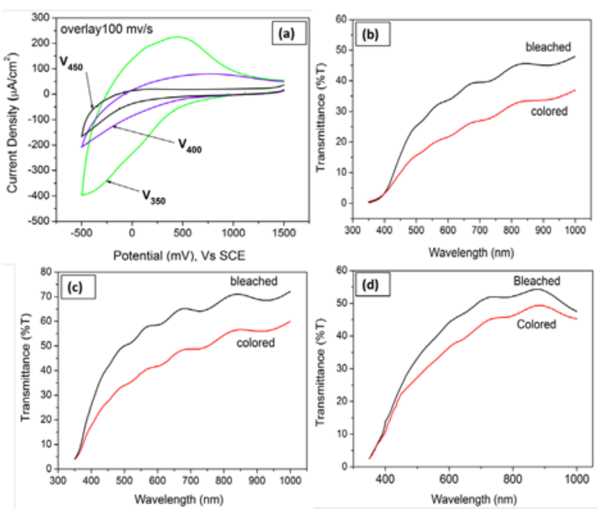

FIGURE2. (a) Overlay of cyclic voltammetry curves of samples V_{350}, V_{400} and V_{450} at scan rate of 100mV/sec. An optical transmittance spectra of the coloured and bleached for (b) V_{350}, (c) V_{400} and (d) V_{450} samples, respectively

Fig.2(a) shows an overlay of the CV recorded for the all samples at the scan rate of 100mV/s. V_2O_5 thin films were colored and bleached in a 0.5 M $LiClO_4$+PC electrolyte by applying a step potential of +1.5 to -0.5V for a fixed time. As the substrate temperature is increased the films becomes more compact, which is not favorable for easy insertion and extraction of ions during coloring and bleaching process. Optical transmittance spectra of the colored and bleached samples were recorded in the wavelength range 350-1000 nm is shown in Fig.2 (b, c, d).

Coloration efficiency (CE), was calculated at 630 nm using following relation,

$$CE_{(\lambda=630nm)} = \frac{\Delta OD}{Q_i} \quad \text{-------------- (6)}$$

The calculated CE varies between 10.29 and 15.16 $cm^2\ C^{-1}$. The CE depends on several preparative parameters of the techniques employed for the deposition. In our case, the CE was maximum 15.16 $cm^2\ C^{-1}$ for V_{350} sample.

CONCLUSION

Polycrystalline V_2O_5 samples with tetragonal crystal structure were prepared at substrate temperatures over 350 to 450°C using a novel PSPT technique. SEM reveals rough and overgrown surface for lower temperatures and compact morphology at 450^{o}C. As the substrate temperature is increased the films becomes more compact, which is not favorable for easy insertion and extraction of ions during coloring and bleaching process. The maximum coloration efficiency and diffusion coefficient for the V_2O_5 thin film deposited at 350^{o}C is found to be 15.16 $cm^2\ C^{-1}$ and 1.13 x 10^{-17} $cm^2\ s^{-1}$, respectively.

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