CHARACTERIZATION OF V_2O_5 AND V_2O_5: Zr OXIDE FILMS PREPARED BY SOL-GEL PROCESS

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Abstract

Pure and zirconium-doped (5 and 10 % mol of Zr) vanadium pentoxide (V_2O_5 and V_2O_5:Zr) thin films were prepared using sol-gel process. The coating solutions were prepared from vanadium (V) oxytripropoxide and zirconium (IV) propoxide as precursors and anhydrous isopropanol as solvent. The films were deposited by dip-coating technique on ITO-Delta glass with a rate of 15 cm/min and next heat-treated at 300 ^oC during 1h. The films samples were characterized by electrochemical and optical measurements. The morphology of the films was studied by scan electronic microscopic (SEM) and atomic force microscopy (AFM). The xereogels of V_2O_5 and V_2O_5:Zr also were characterized by X-ray diffraction (XRD) and thermal analysis (DSC/TGA). The feasibility for use of these films as ion storage electrodes for electrochromic devices was investigated.

Introduction

Vanadium pentoxide films for electrochemical charge storage devices have received significant attention over the last two decades. These films have been extensively studied due to their broad industrial applications, especially in optical switching devices [1]. Moreover, vanadium pentoxide films are also being used as humidity sensors, secondary Li batteries and electrochromic materials [2]. The relationship between microscopic and macroscopic properties, materials, and deposition parameters provides an important guidance to optimize these material characteristics for a given application [3]. This is especially true for vanadium oxides, since these films exhibit very different electrical and optical properties depending on the deposition technique. Vanadium pentoxide films can be deposited by the sol-gel methodology, which offers the advantages of large area deposition, controlled film microstructure and production of films containing multiple cations [4].

Experimental

The starting solution to produce V_2O_5 and V_2O_5:Zr thin films was prepared by mixing vanadium (V) oxytripropoxide in an anhydrous isopropyl alcohol as solvent and glacial

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acetic acid. This mixture was stirred for 30 min. Aiming to obtain V_2O_5:Zr sols, the final solution was doped with 5 and 10 % mol of zirconium (IV) propoxide. The films were deposited by dip-coating method at speed of 15 cm/min. The films were subsequently dried in air at room temperature and next thermal-treated at 300 ^oC for 1hr. Electrochemical measurements were performed using 1M $LiClO_4$/PC as electrolyte. *In situ* visible spectra of the films were measured by an Agilent spectrophotometer.

Results

Fig. 1 shows the cyclic voltammograms and transmittance data of V_2O_5:Zr (5% mol of Zr) thin film. As can be observed in Figure 1a the fresh sample (as deposited) was almost transparent reaching the values of about 80% of transmittance in the range from 600 to 1100 nm. The change in applied voltage from +0.85V to -0.85V promote an decrease in the transmission values to 60% in the range from 400 to 600 nm and about 40% from 600 to 1100 nm and appearance of blue color. At the same time it can be observed in Figure 1b an increase in the cathodic current when a more negative potential was applied indicating the oxide reduction followed by lithium ions intercalation. During the deintercalation process an anodic peak potential at +04V is observed and the transmittance measurements show the recuperation of initial characteristics of the film (Figure 1a), which indicate the reversibility of the intercalation/deintercalation process.

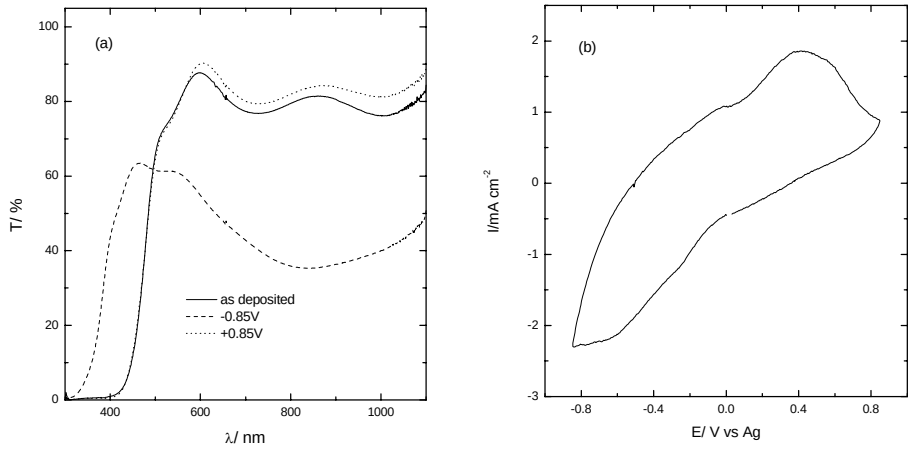

Fig. 1 *Transmittance (a) and cyclic voltammetry (b) measurements of V_2O_5:Zr film.*

Conclusions

In this work, we focused our attention on the electrochromic properties of V_2O_5 thin film and the influence of zirconium on the ion storage capacity. The charge density for V_2O_5:Zr was 51 mC/cm^2 and the film became blue color after lithium electrochemical insertion process. The transmittance variation at λ= 550 nm was 20% and at λ= 800 was 40% between colored and bleached states. Comparing pure and Zr doped V_2O_5 thin films a significant improvement in the ion storage capacity and cycling stability was observed. In

all cases the anodic/cathodic process were reversible and all the results show that V_2O_5:Zr film can be used as a counter electrode in electrochromic devices.

Acknowledgments

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