

Effects of Some Parameters on WO₃ Nanostructures Synthesized by Spin Coating Technique

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Abstract: In this study, WO₃ nanostructures were synthesized on ITO and FTO glasses by Spin Coating Technique at 3000 and 6000 rpm rotational speeds. Effects of substrate and rotational speed on the nanostructures were investigated. Structural, morphological and electrochromic properties of WO₃ nanostructures were characterized by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Cyclic Voltammetry (CV) measurements respectively. It was obtained monoclinic crystal structure, smooth and uniform surface properties for all of the samples. However, it was observed that ITO substrate and low rotational speeds at spin coating technique is more suitable than FTO substrate and high rotational speeds to achieve WO₃ nanostructures with highly electrochromic performance.

Keywords: WO₃ nanostructures spin coating, FTO, ITO

I. INTRODUCTION

Electrochromic (EC) materials have changing optical properties between coloured and bleached state with applied an external voltage or current. [1]. Among them, Tungsten oxide (WO₃) is most important inorganic electrochromic metal oxide [2]. WO₃ changes from colourless to blue when electrons, ions such as H⁺ or Li⁺, are intercalated in its structure with applied electric potential and It reverts to colourless when electrons, ions are de-intercalated from the structure [3]. WO₃ structures are deposited using various methods as a CVD [4], anodic oxidation [5], sputtering [6], spray pyrolysis [7] sol-gel-spin coating [8-9]. Sol-gel method is an attractive route used for the deposition of the films due to its simplicity, safety, easy of doping, high homogeneity, relatively low process temperature and facile control of the product chemical composition [10].

In this study, WO₃ nanostructures were synthesized by sol-gel spin coating technique at two rotation speeds and two type TCO coated (ITO, FTO) substrates.

II. EXPERIMENTAL DETAILS

In the experiment, WO₃ nanostructures were synthesized by Spin Coating technique on FTO and ITO coated substrate at 3000 and 6000 rpm rotation speeds. The spin precursor solution was prepared using Hydrogen Peroxide (26%), tungsten powder (99% purity) and ethanol in an ice bath. The prepared solution was aged 2 month for stability. Aged solution was dropped on FTO and ITO substrates for spin coating process and the substrates were rotated about 30 seconds for each rotation speed. After, the deposition process, the substrates were exposed to heat treatment at 150-200° C for 10-15 minutes. The deposition and heat processes were repeated 5 times to achieve the desired film thickness. Finally, samples were annealed at 500 °C for 2 h in air atmosphere.

The properties WO₃ nanostructures were characterized by XRD, SEM and CV measurements.

A Panalytical Empyrean X-ray diffractometer (45 kV, 40 mA with CuK α radiation $\lambda = 1.5406 \text{ \AA}$) was used to determine structural properties of samples. A FEI Quanta 550 FESEM was used to identify morphological properties of samples and Gamry Model potentiostat were used to analysis EC properties of samples with cyclic voltamograms measurements.

III. RESULTS AND DISCUSSION

Tungsten oxide nanostructures were synthesized at 3000 and 6000 rotation speed by using the spin coating technique. The samples were exposed to annealing treatment. XRD patterns are shown in Fig. 1 for all the samples. The peaks all of the samples matched with monoclinic WO₃ (PDF#43-1035) crystal structure. It is clear that WO₃/ITO samples are more intense peak than WO₃/FTO samples and WO₃ sample that was synthesized at 6000 rpm rotation speed, have more intense peaks than the sample was synthesized at 3000 rpm rotation speed.

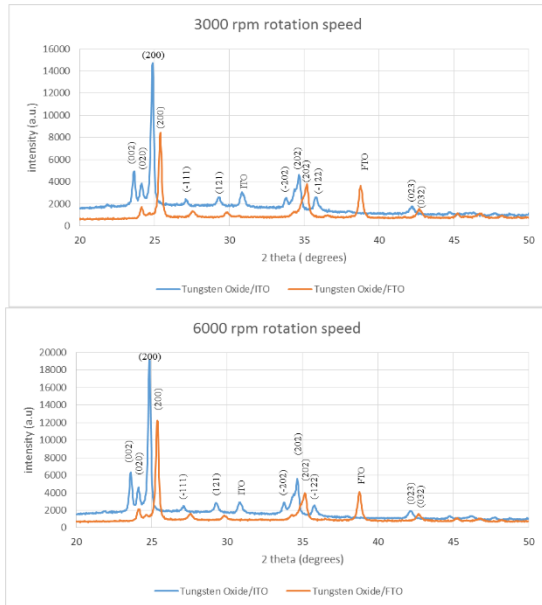


Figure. 1 XRD spectra of WO₃ nanostructures.

SEM images are shown in Fig. 2 for the all samples. It is seen that homogeneous, smooth surfaces for all of the samples at SEM microphotographs of Tungsten Oxide nanostructures on FTO and ITO substrates. However, it is clear that WO₃/FTO samples have more roughness than WO₃/ITO samples. Surface of the each sample is coated with grains at nanoscale along the surface.

Curves of CV measurements are shown in Fig. 3 for all of the samples. It is characterized electrochromic properties of Tungsten Oxide nanostructures by spectral characterization (at -2V and +2V range) using Cycling Voltammetry with scanning rate of 500 mV/s. It is clear that WO₃/ITO samples show better electrochromic feature than WO₃/FTO samples. During CV measurements, the following reaction occurs:



During coloration, WO₃ samples turns blue colour, when reverse voltage is applied, colour of samples is bleached. WO₃/ITO (3000 rpm) sample is larger curve area than WO₃/ITO (6000 rpm). Therefore, WO₃/ITO (3000 rpm) sample shows better electrochromic performance than other. Besides, WO₃/ITO samples show better electrochromic performance than WO₃/FTO samples according to CV curve area of the samples. In addition to the results, Fig. 4 shows images of the WO₃/ITO samples turned blue colour.

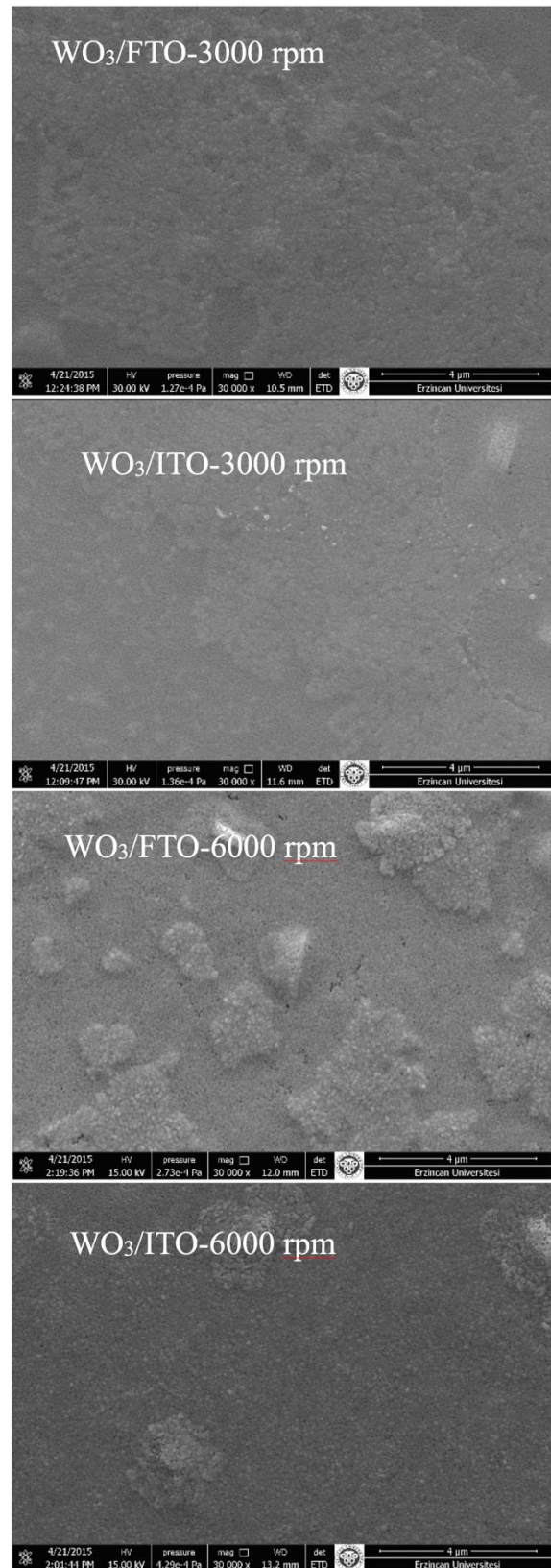


Figure. 2 SEM images of WO₃ nanostructures.

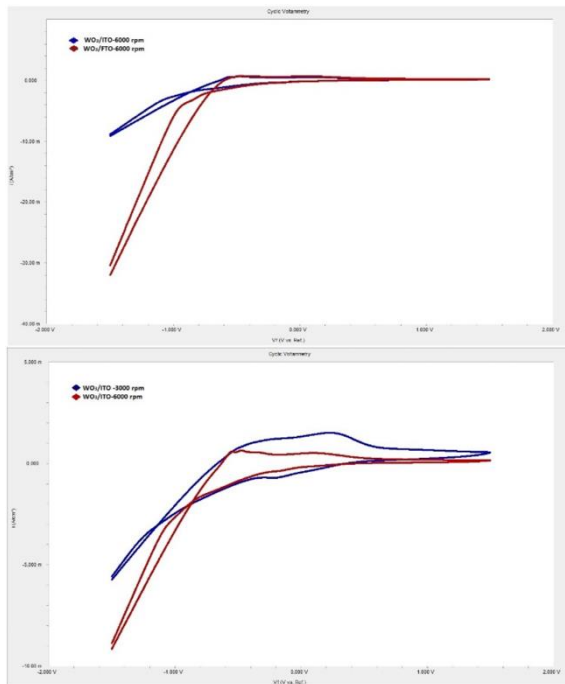


Figure. 3 CV curves of WO₃ nanostructures.



Figure. 4. Photographs of the Electrochromic WO₃/ITO samples turned blue colour synthesized with Spin Coating at 3000 rpm and 6000 rpm respectively.

IV. CONCLUSIONS

As a result, in this study, WO₃/ITO and WO₃/FTO nanostructures were synthesized by using simple solution-based economical Spin Coating Method at two type rotation speed and substrate. Properties of the nanostructures were analysed by using XRD, SEM and CV measurements. According to results, all of the samples show the electrochromic performance that may be used in electrochromic applications. However, it is obvious that WO₃/ITO (3000 rpm) has better electrochromic performance than others.

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