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Electrochemical properties of TiO₂ electrode prepared by various methods

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Abstract

Thin transparent TiO₂ layers were created on the conductive ITO glass by means of the templated sol-gel technique and by the subsequent calcination at 450° C. The sol-gel method using molecular templating is based on a chemical process utilizing hydrolysis and polycondensation of metal alkoxides in the core of reverse micelles which allows a production of uniform particles in layers. The sol-gel method was chosen by reason of the sol-gel layers electrodes are transparent and possess very stable surface. For the preparation of the thin sol-gel TiO₂ films, numerous deposition techniques were applied. This contribution is focused on the study of structural and photo-electrochemical properties of the sol-gel nanostructured layers deposited by two various techniques (a dip-coating and an inkjet printing). The sol's viscosity, concentration, solvent volatility, speed of pulling etc. may influence the final structural properties of layers, such as film thickness, nanoparticles size and surface morphology. The surface properties were determined by XRD, Raman, SEM, AFM and UV-Vis analyses. Photo-induced electrochemical properties were measured by potentiodynamic methods in the three-compartment electrochemical cell. This Pyrex cell contained supporting electrolyte (0.1M Na₂SO₄) and the TiO₂/ITO electrode was used as a working electrode. As an UV source the polychromatic mercury lamp was employed and the wavelength of the incident light was focused by an interference filter on 365 nm.

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1. Introduction

Nanoscaled titanium dioxide (TiO_2) in thin layer or powder forms appertains to the most extensively studied semiconductors. This metal oxide is a promising semiconductor frequently used as a photocatalyst in the advanced oxidation processes (water or air purification) due to its non-toxicity, chemical stability, photocatalytic activity and low cost [1-4]. Especially, thin films as the nanostructured electrode materials have become very important in fields of photovoltaics, energy storage, sensing, photoelectrocatalysis etc. [5,6]. As generally known, the photoactivity arises from the semiconductor nature, especially from the ability of the light quantum absorption followed by the charge carrier generation. TiO_2 in crystallographic form of anatase has become an interesting candidate as an n-type photoanode due to its excellent efficiency to generate the electron-hole pairs [7,8].

Preparation of the nanostructured electrode materials with highly uniform nanoparticles has been investigated by many groups [9-11]. The most commonly used method is the sol-gel technique utilizing the molecular templates. The main advantage of this purely chemical method lies in a possibility of layer preparation under laboratory conditions as well as the possibility to tailor TiO_2 layer properties by varying preparation conditions. Nanoparticles with controlled chemical composition, size distribution, uniformity and dispersion can be readily synthesized using reverse micelles [12-14]. Layers prepared in this way possess usually highly transparency, surface homogeneity, size, structure uniformity and very high adhesion to substrate in comparison with the particulate layers.

Nowadays, a variety of deposition techniques for the liquids deposition, such as dip-, spin- or spray-coating, doctor blade, roller etc., has been appeared and each of them offers some advantages and disadvantages [15]. There are three traditional methods of thin layer preparation from liquid precursors: spin-coating, spray-coating and dip-coating. Dip-coating belongs to the traditional and widely used methods of the thin layer preparation. It is based on dipping the substrate into the sol and pulling it out at constant speed. Spray-coating is the other elementary technique which applies liquid sol on the substrate by the spray head with one three-axe system nozzle. And the last method spin-coating uses centrifugal force to form a film of liquid precursor [15-17]. As a new process of the sol application the ink-jet printing has been appeared. This technique proved to be very elegant and clean method for liquids deposition and direct patterning [18].

The sol viscosity, concentration, solvent volatility and the speed of pulling influence the resulting film thickness and structural properties. This contribution is focused on the study and the comparison of structural and photo-electrochemical properties of the sol-gel nanostructured TiO_2/ITO electrode deposited by dip-coating and inkjet printing.

2. Experimental

2.1. Layers preparation

Thin transparent TiO_2 layers were created on the one side of the conductive ITO glass (5–15 Ω , Delta-Technologies Ltd., USA) and on the soda-lime microscopic glass plates by means of the templated sol-gel technique and by the subsequent calcination at 450° C for 4 h. The sol-gel method using molecular templating is based on a chemical process utilizing hydrolysis and polycondensation of metal alkoxides in the core of reverse micelles which allows a production of uniform particles in layers. The reverse micelles were created by molecular templates TX 102 (Sigma-Aldrich) in the nonpolar environment of cyclohexane (Aldrich, 99.9%, HPLC grade) or xylene (p.a., Penta, Czech Republic). In the core of the created micelles a small amount of demineralised water was added. As titan precursor the titanium

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