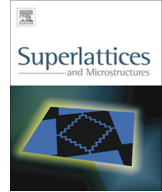




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# Synthesis of TiO<sub>2</sub> nanotube array thin films and determination of the optical constants using transmittance data



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## ABSTRACT

TiO<sub>2</sub> nanotube arrays were grown on glass substrate by ZnO nanorod sol–gel template process. XRD analysis and FESEM microscopy were employed to characterize the structural and morphological properties of the prepared nanotube. EDX and UV–Vis spectroscopy were used to assess the chemical composition and study the optical properties of the film. An optical model has been performed to simulate the optical constants and thicknesses of the films from transmittance data using the Levenberg–Marquardt algorithm via Drude model. The simulated transmittance is in good agreement with the measured spectrum in the whole measurement wavelength range. The refractive index and extinction coefficient, thickness and dielectric function of TiO<sub>2</sub> nanotube films were calculated by Drude model. Also, the related absorption coefficient, optical bandgap and porosity were determined.

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## 1. Introduction

In recent several decades, there has been increasing interest in one-dimensional nanostructures (nanorods, nanowires, nanotubes) because of numerous potential applications in various areas such as photocatalysis, solar energy, electronics, optics and sensor [1–6].

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Among the various inorganic materials, the TiO<sub>2</sub>-based one-dimensional structures have attracted much attention for potential nanoscale electronics, optoelectronics and biochemical-sensing applications due to its high refractive index, chemical stability, and superior photocatalytic activity [7–10].

At present, one-dimensional TiO<sub>2</sub> nano arrays can be prepared by many methods, including template-assistant approaches [11], direct oxidation of Ti substrate [12], electrochemical anodization of titanium [13], metal–organic chemical vapor deposition [14], and hydrothermal synthesis [15].

Among these techniques, the sol–gel method presents many advantages, such as simple equipment and low cost, high optical quality of films, and low crystallization temperature [16]. The sol–gel template method has been reported to synthesize TiO<sub>2</sub> nanotubes, nanorods and nanowires from the anodic alumina membranes, organogel and supramolecular template. Lei miao et al., reported TiO<sub>2</sub> nanorods by a heating–sol–gel template process [17].

Qiu et al. [18,19] fabricated TiO<sub>2</sub> nanotube arrays by using an aqueous solution synthesized ZnO rod as a template. Seok-In Na and co-workers used electrodeposited ZnO nanorods as a template for the fabrication of TiO<sub>2</sub> nanotubes [20]. In this study, ZnO nanorods have been used as a template for fabricating TiO<sub>2</sub> nanotubes.

Commonly, the optical constants are retrieved by simulating the transmittance based on dielectric models and using a nonlinear least squares fitting procedure [21]. The Drude model [22] has often been applied to investigate the optical properties of ITO films. Frequently, the Drude model performs the simulation, together with other optical models, such as the Tauc–Lorentz model [23], Lorentz oscillator model [24–27], Forouhi–Bloomer model [28], and Bruggeman effective medium approximation [29,30]. Brewer and Franzen [31] used the Drude model with a constant relaxation time to fit the variable angle reflectance FTIR spectra of ITO and FTO films in the mid-IR range.

The Levenberg–Marquardt method is an iterative algorithm for solving nonlinear least squares problems. Least-squares problems minimize the difference between a set of data and a model function that approximates these data [32].

By application of Levenberg–Marquardt least square method, the experimental transmittance data were fitted completely with the transmittance data calculated via Drude model. Herein, we perform a minimizing method by the well-known Levenberg–Marquardt algorithm [33]. For this purpose, several constraints for data fitting and prediction of optical constants for TiO<sub>2</sub> films are applied using transmittance data.

## 2. Experimental

### 2.1. Synthesis of ZnO seed layers and growth of ZnO nanorod arrays

Initially, 0.5 g of Zn(Ac)<sub>2</sub>·2H<sub>2</sub>O was dissolved in 100 ml ethylene glycol (C<sub>2</sub>H<sub>6</sub>O<sub>2</sub>) and 5 ml deionized water and then stirred for 30 min at 140 °C on heater to obtain a stable and homogeneous solution. Glass was used as the substrate and cleaned by sonication in acetone, ethanol and deionized water, prior to use. The cleaned glass substrates immersed into ZnO sol at the rate of 3 cm/min. Subsequently, the coated substrates were dried in air and annealed at 150 °C for 1 h in muffle furnace. This process was repeated for at least three times to produce ZnO seed layers. Vertical nanorod arrays were grown by immersing the seeded substrates in aqueous solutions of 0.05 g zinc nitrate tetra hydrate and 0.8 g Natriumhydroxid into 200 ml deionized water for 1 h at 70 °C. Finally, the substrates were dried in air.

### 2.2. Fabrication of TiO<sub>2</sub> nanotube arrays

The process for the formation of TiO<sub>2</sub>-covered ZnO nanorod arrays onto glass substrates was performed with the Ti-precursor composed of 2 ml Ti isopropoxide in 150 ml 2-propanol solution containing 0.75 ml HCl. The substrate with ZnO nanorods was dipcoated into Ti-precursor solution, followed by heat treatment at 450 °C for 30 min in a furnace to form the TiO<sub>2</sub> crystalline structures. Afterward, the substrates with TiO<sub>2</sub>-covered ZnO nanorods were immersed into an aqueous solution of 0.75 M HCl to remove the ZnO nanorod template. After the immersion in HCl solution for 5 min at room temperature, all ZnO was etched out and only the TiO<sub>2</sub> nanotubes remained.

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