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Deposition of porous titanium oxide thin films as anti-fogging and anti-reflecting medium

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a r t i c l e i n f o

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A B S T R A C T

High transparent TiO₂ thin film has been deposited by a dip-coating method. The synthesized sol was obtained through the hydrolysis of titanium iso-propoxide under the selected pH. The X-ray diffraction (XRD) results show amorphous thin films. The transmission through films was characterized by spectrophotometer.According to transmission spectra offilms, band gap, skin depth, Urbach energy, refractive index and extinction coefficient has been determined. The optical band gap of the films has been estimated to be in the range from 3.38 to 3.09 eV. Scanning Electron Microscopy (SEM) has been carried out for morphology characterization of the films surface which shows dense surface of films with few cracks on the surface.

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1. Introduction

The wide-band-gap titanium oxide material has widely been investigated because of its remarkable optical and chemical properties. One promising application of $TiO₂$ is the self-cleaning properties of glasses in automotive industry. In hydrophilic functions rainwater hits the glass and spreads evenly and wipe off the loosened dirt with it, also drying instantaneously without producing streaks $[1]$. In the photo-catalytic function there is a high recombination rate of photo-induced electron–holes in the film that shift to the surface and interact with daylight to break apart the organic dirt $[2]$. Self-cleaning photo-catalytic TiO₂ films are beneficial since they reduce the maintenance cost and enhance the efficiency of various optical systems, especially thermal and photovoltaic solar systems.

Among all the available deposition techniques, the sol–gel technique is a versatile method to deposit metal oxide thin films at low temperatures, with appropriate homogeneity, control over composition and low cost. The dip-coating technique is a suitable method to deposit anti-reflection and self-cleaning films of $TiO₂ [3]$.

The focus of the present work is to synthesize $TiO₂$ thin films via sol–gel dip coating technique and annealing films at five different temperatures between 80 and 120 ◦C. Afterwards films were characterized using techniques like XRD, SEM, Fourier transform infra

[http://dx.doi.org/10.1016/j.ijleo.2016.03.009](dx.doi.org/10.1016/j.ijleo.2016.03.009) 0030-4026/© 2016 Elsevier GmbH. All rights reserved. red spectroscopy (FTIR), UV–vis–NIR spectrophotometer and ellipsometry. The effects of annealing temperatures on the thickness, phase formation, surface morphology and optical properties of the films were investigated and correlated.

2. Experimental details

Sol of titanium dioxide was synthesized using titanium tetra iso prop-oxide (TTIP), iso-propanol (i-PrOH), de-ionized water and sulfuric acid (H_2SO_4) . In i-PrOH, TTIP was added, and then the resulting sol was stirred at 500 RPM at room temperature (27 ◦C). After 2 h a solution consisting of i-PrOH, H_2SO_4 and DI water was added drop wise to prepared sol and was stirred at 500 RPM at room temperature. pH of sol was 4. Sol was then aged for 48 h for polymerization and densification. A transparent sol with white nano-particles of titanium dioxide was obtained. The thin films of titanium dioxide were deposited by dip-coating on the CR-39 at constant withdrawal speed of substrate 250 mm/s. Then the heat treatment was given to films at five different temperatures (80 ◦C, 90 ◦C, 100 ◦C, 110 ◦C and 120 \degree C) to check the effect of annealing temperature on the characteristic of thin films.

The size distribution and grain size of thin films were calculated by analyzing the SEM image using SEM S-3400N Hitachi. The optical transmission of thin films was measured using a UV/VIS/NIR spectrophotometer (HITACHI U-2800) within a wavelength range of 200–1100 nm and ellipsometer (J.A. Woollam, Spectroscopic Ellipsometer). The crystal structure of $TiO₂$ thin films was examined by performing withX-ray diffractometer (Bruker, D8-Advanced) using

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CuK α radiation with a scanning rate of 4°/min in the 2 θ range of 20–90◦. Chemical composition and chemical bonding were determined by FTIR Model M 2000 Midac USA spectrophotometer.

3. Results and discussion

3.1. FTIR study

Fourier transform infrared (FTIR) spectrum analysis of dip coated TiO₂ thin films is shown in Fig. 1. Absorption bands observed at 615 and 472 cm⁻¹ correspond to Ti–O vibrations [\[4\].](#page--1-0) The bands at 1321 cm⁻¹ and 1132 cm⁻¹ corresponds to C–O vibrations. Band observed at 1635 cm⁻¹ corresponds to the C=O vibrations [\[5\].](#page--1-0)

3.2. Structural studies

X-ray diffraction (XRD) patterns of all the five films dip coated on CR-39 at withdrawal speed of 250 mm/s were obtained as represented in Fig. 2. Films were annealed at five different temperatures ranging from 80 to 120 ℃. The X-ray diffraction patterns of all titanium dioxide thin films show same trend. These patterns indicate that films are amorphous. It was a known fact that crystallization occurs by annealing at appropriate higher temperatures otherwise material remains amorphous. Substrate CR-39 can with stand temperature up to 130° C. So the limited heat treatment temperature resulted in amorphous structure of the films.

3.3. Optical properties measurements

Fig. 3 presents the transmittance spectra of the $TiO₂$ films after thermal annealing. The transmittance of the films decreased weakly as the annealing temperature increased from 80 to 120 ◦C [\(Table](#page--1-0) 1).

Fig. 1. FTIR spectra of the TiO₂.

Fig. 2. XRD pattern of TiO₂ thin films annealed at temperature (a) 80, (b) 90, (c) 100, (d) 110 and (e) $120 °C$.

Fig. 3. Transmission spectra of TiO₂ thin films annealed at temperature (a) 80, (b) 90, (c) 100, (d) 110 and (e) 120 °C.

In this work, the optical thickness was calculated by the envelop method [\[6\]](#page--1-0) for the annealed films. From the transmission curve thickness of the films were calculated using the following relation:

$$
t = \frac{M\lambda_1\lambda_2}{2[n_1\lambda_2 - n_2\lambda_1]}
$$

where n_1 and n_2 are the refractive indices at the two adjacent maxima (or minima) at λ_1 and λ_2 and t is the thickness of the thin films and thickness was found to be in the range of $1.9-2.9 \,\mu m$. From the optical measurements, it is observed that thickness of the TiO₂ films increases with increase in annealing temperatures. The films have a substantial transmission in the range 500–900 nm. It is found from Fig. 2 that the average transmittance of all films is about 83–88% in the visible region. Films annealed at low temperatures seem to have slightly better transmittance in the visible range. It may be due to the less light scattering effect of its lower surface roughness.

The refractive index was calculated from the following relation

$$
n = [N + (N^2 - n_0^2 n_1^2)^{1/2}]^{1/2}
$$

where

$$
N = \frac{n_0^2 + n_1^2}{2} + \frac{2n_0n_1(T_{\text{max}} - T_{\text{min}})}{T_{\text{max}} * T_{\text{min}}}
$$

$$
n_0 = 1.0; n_1 = 1.498.
$$

 T_{max} and T_{min} are maximum and minimum transmittances at the same wavelength in the fitted envelope curves on the transmittance spectrum. Using the envelop $[6]$ method, the refractive index n of the $TiO₂$ films for different annealing temperatures was calculated. It is observed that there is only a marginal increase in refractive index with annealing. In this project, the small increase of refractive index was due to the densification and compactness of the films at increasing annealing temperatures.

The absorption coefficient a of $TiO₂$ films was determined from transmittance measurements

$$
\alpha = \frac{1}{t} * \ln \left[\frac{2R2T}{-(1-R)^2} + \sqrt{(1-R)}4 + 4R2T2 \right]
$$

These absorption coefficients values were used to evaluate optical energy gap. To quantify the direct optical band gap of the films, $(\alpha h \nu)^2$ vs hv and for indirect band gap $(\alpha h \nu)^{1/2}$ vs hv were plotted for the TiO₂ films at different annealing temperatures. It was observed that the direct optical band gap for $TiO₂$ films decreased from 3.38 to 3.09 eV and indirect band gap decreased from 2.7 to 2.36 eV as the annealing temperature increased. This band gap range shows that rutile phase of titania has been achieved although annealing temperature is quite low for rutile phase formation [\[7,8\].](#page--1-0) Band gap is decreasing with increase in annealing temperature Download English Version:

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