Fabrication of Transparent Titanium Dioxide Thin Film at Low Temperature by Sol-gel Spin Coating Method

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Abstract—Titanium Dioxide (TiO2) thin film with various sol-gel concentrations has been successfully prepared using sol-gel spin coating method. The preparation of the TiO2 thin films was performed at room temperature. The electrical and optical properties were characterized using 2-point probe I-V measurement and UV-Vis-NIR Spectrophotometer. For electrical properties, 0.2M of sol-gel concentration gives the lowest sheet resistance among other concentrated sol-gels. The optical transmission in the visible region (450–1000 nm) for 0.1M and 0.2M are the highest (>80%), indicating that the films are transparent in the visible region. The transmission decreases sharply near the ultraviolet region due to the band gap absorption. The prepared TiO2 thin film can be used in many applications such as solar cell application, sensors and Ultra Violet coatings.

I. INTRODUCTION

Titanium dioxide (TiO2) films have been largely studied as photoanodes in the process of photo electroysis of water in solar energy conversion systems [1–3] and electrochromic materials for display devices [3,4]. Other technological applications of TiO2 coatings include its prospective use as a material for smart windows [5], antireflective coatings [6,7], optical filters [8], in catalysis and solar cells. TiO2 films can be prepared using various kind of methods: thermal [9] or anodic [10] oxidation of titanium, electron beam evaporation [11], ion sputtering [12], chemical vapour deposition [13], including plasma-enhanced chemical vapour deposition [14], and the sol–gel method [15, 16]. Since 1972, researchers have been study [17], titanium dioxide as one of the most important electrode materials for semiconductor electrochemistry [18].

At normal pressure and temperature, three different TiO2 crystalline structures are stable: rutile, anatase, and brookite. Nowadays, researcher had been taken a step forward to study titanium dioxide thin film for coating, solar cell and electronic application. The characteristics of the films are generally affected by the preparation conditions such as the deposition method, post deposition annealing temperature and types of substrates. It has already been observed that the optoelectronic properties of sol–gel TiO2 films are influenced largely by the thickness of the film [19]. Especially, for a polycrystalline film, the grain boundary-related micropores which depend on the film thickness control the photoconduction properties. For a sol–gel film, the thickness can be varied by the number of coatings and also by the concentration of the sol. The latter affect the thickness via the viscous drag on the substrate by the moving liquid. In this paper, we have investigated the effect of sol concentration on the surface morphology, structural properties and optical properties of the TiO2 thin film grown on silicon and glass substrates by a sol–gel and deposited using spin coating technique. Sol-gel concentrations that we used are 0.1M, 0.2M, 0.3M and 0.4M.

II. EXPERIMENT

a. Preparation of nanostructured TiO2 thin film

The glass substrate was used as substrates. The glass were cleaned with acetone, methanol and distilled water, followed by drying in oven. Substrates were cleaned by the ultrasonically method. TiO2 thin films were deposited onto glass substrates using sol–gel, spin coating method. Different sols of concentration 0.1M, 0.2M, 0.3M, and 0.4 M were prepared by adding required amount of Titanium (IV) Isopropoxide [Ti (OC3H7)4] to ethanol. Triton X-100 was added to the solution as a sol stabilizer followed by a thorough mixing process with a magnetic stirrer. Glacial Acetic acid was added at chelating agent to the sol-gel. To complete the reaction in the sol-gel, DI water was added while stirring process is done. Nanopowder Titanium (IV) oxide (Aldrich, 99.9%) were introduced into the sol-gel.
which is react as nanofiller to the sol-gel that can improve the conductivity of thin film. All the chemicals used were of AR grade and were used as received without further purification. The sol-gels were heated at 50°C for 1h, using magnetic stirrer and then were continue stir for 3h at room temperature. Heating process is to increase the reaction process between the materials in the solution. The TiO₂ thin films were deposited onto glass substrates by using spin coating technique. Spin coating was set at a withdrawal speed of 3000 rpm and for 30 seconds. The nitrogen (N₂) gas was supply at 60 psi. The TiO₂ solution was dropped ten times onto the substrates and then dried the TiO₂ thin films in the oven at 100°C for 5 minutes. The drop and dry process was repeated for five times onto the same substrate. Finally, the films were annealed at 500°C for 1 hour in the furnace to obtain the TiO₂ thin film without any gases. The thicknesses of the TiO₂ thin films were measured by Scanning Electron Microscopy (JEOL JSM-6380LA).

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**b. Characterization method**

Optical transmittance properties were studied using VARIAN 5000 UV-Vis-NIR Spectrophotometer from 200 nm to 1000 nm of wavelength. The IV measurement was performed using 2 points probe, using a (Advantest R6243) source measuring unit. Fig. 1 shows diagram of coated Au on top of TiO₂ thin film which act a metal contact to perform I-V measurement. 2-point probe will be connected at this metal contact.

![Diagram of Au coating process on glass substrate](image)

**III. RESULTS AND DISCUSSION**

**a. Electrical properties study**

Current-Voltage (I-V) measurements were performed to the TiO₂ thin films structure to determine their electrical properties. Current versus voltage graph has been plotted and it shows that 0.2M of sol-gel gives the lowest resistance among the other concentrated sol-gels. The resistance of the thin film decreased from 0.1M to 0.2M of sol-gel. But resistance of this prepared thin film starting increased when it became 0.3M and 0.4M of sol-gel. The reason to the increasing of resistance is due to increasing amount of solute. High amount of solute will affect the electron mobility and will limit the current flow in the film. From this, it can be said that additional of nanofiller such as nanopowder TiO₂, will give good effect to the electron mobility but too many of it, can effected to the electrical properties. It is good alternative besides using ion metal as a dopant to improve conductivity of the thin film, but limitation must be considered.

**b. Optical properties study**

**Transmittance spectra**

The effect of sol concentration on the optical transmittance and the band gap (Eg) values of the TiO₂ thin films have been studied. The transmission spectra of the TiO₂ films are shown in Fig. 4. The optical transmission in the visible region (450–1000 nm) for 0.1M and 0.2M is high (>65%), indicating that the films are transparent in the visible region. The transmission decreases sharply near the ultraviolet region due to the band gap absorption. From the graph, we can observe that 0.3M and 0.4M are not in transparent solution. It is gives only 30% of transmittance percentage. For solar cell application, 0.3M and 0.4M of sol-gel concentration will not give good result as prepared solar cell cannot get all of the visible light.

![Transmission spectra of nanostructured TiO₂ thin films at different concentration](image)

**IV. CONCLUSION**
TiO$_2$ thin film has successfully deposited at different sol-gel concentration, 0.1M, 0.2M, 0.3M and 0.4M respectively. Electrical and optical properties have been studied. For electrical properties, the current intensity increased from 0.1M of sol-gel concentration until 0.3M of concentration but decreased at 0.4M of concentration. It can be concluded that sheet resistance of TiO$_2$ thin film did not affected by the increasing of sol-gel concentration. Only 0.1M and 0.2M gives good transparency compared to 0.3M and 0.4M. By referring to electrical and optical properties of prepared TiO$_2$ thin film, it can be said that prepared TiO$_2$ thin film can be used in solar cell application as n-type semiconductor or as a coating layer. Suitable concentration has to be considered for use in other application.

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