

Deposition of Titanium Dioxide (TiO₂) Thin Films Using In-house Nano-TiO₂ Powder

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Abstract – *The purpose of this study is to fabricate uniform TiO₂ thin films using in-house TiO₂ nanopowder. The nanopowder was obtained from a tin mining waste (Ilmenite) and its concentration was optimized during fabrication. The TiO₂ thin films were characterized by an atomic force microscope (AFM), a surface profiler, an X-ray diffractometer, an Ultra violet- Visible (UV-Vis) and a current-voltage (I-V) measurement system. The relation of the uniformity and the properties of the TiO₂ thin films will be discussed in detail in this paper.*

Keywords: *Titanium dioxide (TiO₂), ilmenite, thin films, sol-gel, atomic force microscope (AFM).*

I. INTRODUCTION

Titanium dioxide or known as TiO₂ has become one of the researched semiconductor material because of the promising result in the photocatalytic oxidation. TiO₂ has wide band gap energy around 3.2 eV or have photon energy ($h\nu$) <390 nm that is transparent to visible light and has excellent optical transmittance [1]. It has high refractive index and good insulating properties, and as a result it is widely used as protective layer for very large scale integrated circuits and for manufactures of optical elements. Besides that, Titanium dioxide is made of TiO₂ octahedral. The structural of octahedral gives three different polymorphs of Titanium dioxide that is anatase, rutile and brookite [2].

Anatase and rutile are the most researched polymorphs but anatase is proven recently as the most photocatalytically active of the three polymorphs. Furthermore, basically anatase transform into rutile under the heat between 600 °C and 700 °C. TiO₂ has many structures but it depends on its polymorphs [3]. If they are anatase and rutile, then the structure is tetragonal but if it is brookite, the structure is orthorhombic [4-5]. Nowadays TiO₂ has wide application from paint to sunscreen to food coloring.

The price of TiO₂ powder in the market for fabrication of TiO₂ thin films is a way too high, but we can bring it into a solution. In this research, TiO₂ powder obtained from tin mining waste (Ilmenite) is introduced which will reduce the

cost up to 100 times. However, it is an issue whether TiO₂ obtained from Ilmenite can be a functionalized material or not. Moreover, it is questionable whether the TiO₂ can be fabricated into a uniform thin film. Using sol-gel, the challenge is to control the process parameters which influence the material properties (electrical and optical). Therefore, process optimization is required to enhance the electrical and optical properties.

II. METHODOLOGY

An aqueous solution was prepared from in-house nano-TiO₂ powder by varying its mass from 1g, 0.4g, 0.1g and 0.01g in 30 ml of ethanol and mixed with 6 ml acetic acid as the catalyst. Then, the solution was stirred on a magnetic stirrer at 700 r.p.m. at room temperature for 20 hours. ITO was cleaned using acetone in ultrasonic cleaner for 5 minutes before rinsed with de-ionized water. The samples were dried under atmospheric ambient at room temperature.

The thin films were fabricated using a spin coater using 2 steps procedure (1000 r.p.m. for 30s and 3000 r.p.m. for 60s). Each layer of the film requires 10 drops of TiO₂ solution and we prepared 10 layers for each sample. Then, the samples were annealed at 500°C for anatase phase. The annealing process was carried out at 1 hour and the samples underwent slow cooling at room temperature at about 5 minutes.

The samples were characterized using an atomic force microscopy (Park System XE-100) for topological observation, a thickness Profiler (KL-Tenko) for thickness measurement, a UV-Vis spectrometer (Varian) for the optical characterization and an I-V measurement system (Keithley 2400) for the electrical measurement.

III. RESULT AND ANALYSIS

The thickness of the sample using mass concentration of 1g, 0.4g, 0.1g and 0.05 g are 427 nm, 57 nm, 54 nm and 17 nm, respectively. Fig.1 (a)-(d) indicates the TiO₂ thin films when the nano-TiO₂ powder concentration was reduced from 1g to 0.05g.

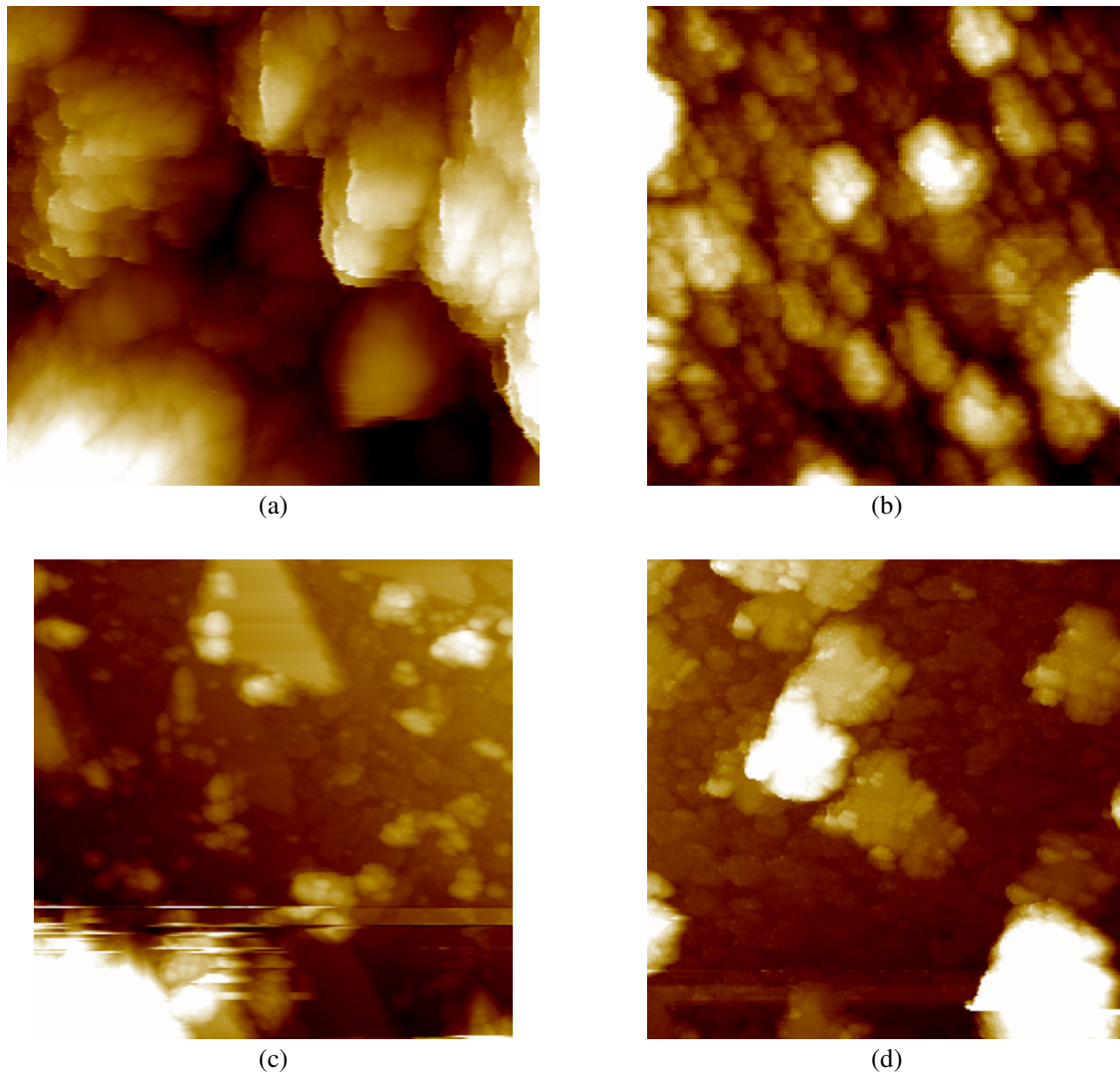


Fig. 1 The AFM topography of TiO_2 thin films by varying the nano- TiO_2 powder mass concentrations (a) 1g; (b) 0.4g; (c) 0.1g; (d) 0.05g

As the concentration of the nano- TiO_2 powder reduced, the particles size which constructs the thin film also reduced. It is observed that TiO_2 thin film deposited using 0.4g has the optimum uniformity.

The transmittance spectra were measured using a UV-Vis spectrometer. Fig. 2 shows the transmittance spectra of the TiO_2 thin films deposited using different nano- TiO_2 powder mass concentration. Generally, TiO_2 has high absorption at wavelength of 360nm due to its optical band gap. As the mass concentration of nano- TiO_2 powder decreased, the optical transmittance increased. There are two reasons of explaining this changes; (i) since the thickness of the film reduced, the transmittance increases, and (ii) as the particles size become smaller, the transmittance became higher. However, the transmittance for 0.4g at wavelength below 400 nm depicted a

different behavior compared to the other samples. The reason is still unknown.

Fig. 4 and 5 show the I - V characteristics of the TiO_2 samples which indicate Schottky and Ohmic response, respectively. The Schottky contact may resulted from the large different of conduction band between Platinum (Pt) and TiO_2 which has the value of 5.6 eV and 4.4 eV, respectively. Therefore, for an electron to be excited from the conduction band of Pt to the conduction band of TiO_2 , it requires energy greater than 1.2 eV. However, this phenomenon is not happening for 0.1g and 0.05g. Instead of Schottky, an Ohmic response was produced. This may be due to reduce in the thickness of the film which causes electron tunneling between the Pt and TiO_2 .

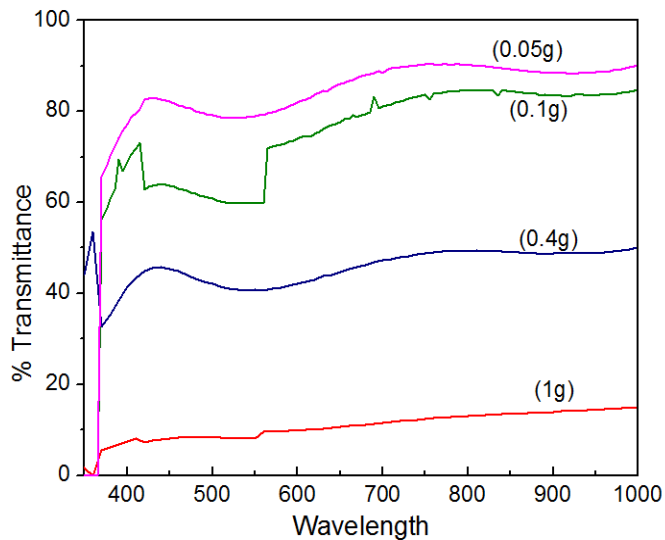


Fig. 2 The transmittance spectra of TiO₂ thin films

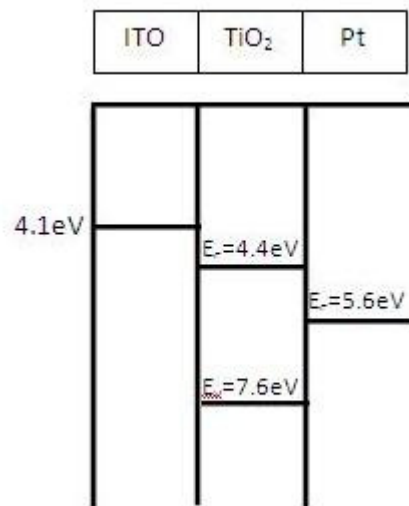


Fig. 3 The band off-set of ITO/TiO₂/Pt contact

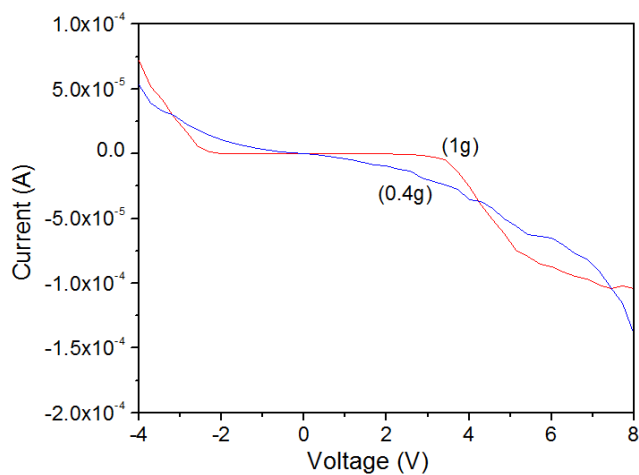


Fig. 4 The Schottky behavior of TiO₂ thin films

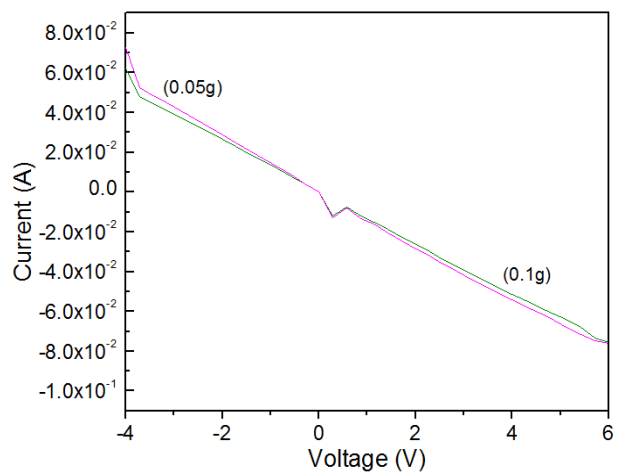


Fig. 5 The Ohmic behavior of TiO₂ thin films

IV. CONCLUSION

As a conclusion, Ilmenite is a good material to fabricate uniform TiO₂ thin films although its purity is only 95.07 %. It can be used as a functionalized material whether for electronic or optoelectronic applications. Furthermore, spin coating is the suitable fabrication technique in sol-gel method. From the characterization analysis, we can conclude that sample deposited using 0.4 g of nano-TiO₂ powder have the best characteristic for both electrical and optical properties. There a few recommendations for a better result. Ethanol is not a good solvent. So, using other different solvent which has higher

solubility may improve the results. The acetic acid which act as a catalyst may also need a process optimization which would produce more uniform thin film.

V. ACKNOWLEDGEMENT

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VI. REFERENCES

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