Deposition of Titanium Dioxide (TiO_2) Thin Films Using In-house Nano-TiO_2 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 TiO_2 has become one of the researched semiconductor material because of the promising result in the photocatalytic oxidation. TiO_2 has wide band gap energy around 3.2 eV or have photon energy (hv) <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 TiO_2 octahedral. The structural of octahedral gives three different polymorphs of Titanium dioxide that is anatase, rutile and brooktile [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 $^{\circ}$C and 700°C. TiO_2 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 brooktile, the structure is orthorhombic [4-5]. Nowadays TiO_2 has wide application from paint to sunscreen to food coloring.

The price of TiO_2 powder in the market for fabrication of TiO_2 thin films is a way too high, but we can bring it into a solution. In this research, TiO_2 powder obtained from tin mining waste (Ilmenite) is introduced which will reduce the cost up to 100 times. However, it is an issue whether TiO_2 obtained from Ilmenite can be a functionalized material or not. Moreover, it is questionable whether the TiO_2 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-TiO_2 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 TiO_2 solution and we prepared 10 layers for each sample. Then, the samples were annealed at 500^{o}C for anatase phase. The annealing process was carried out at 1 hour and he 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 TiO_2 thin films when the nano-TiO_2 powder concentration was reduced from 1g to 0.05g.

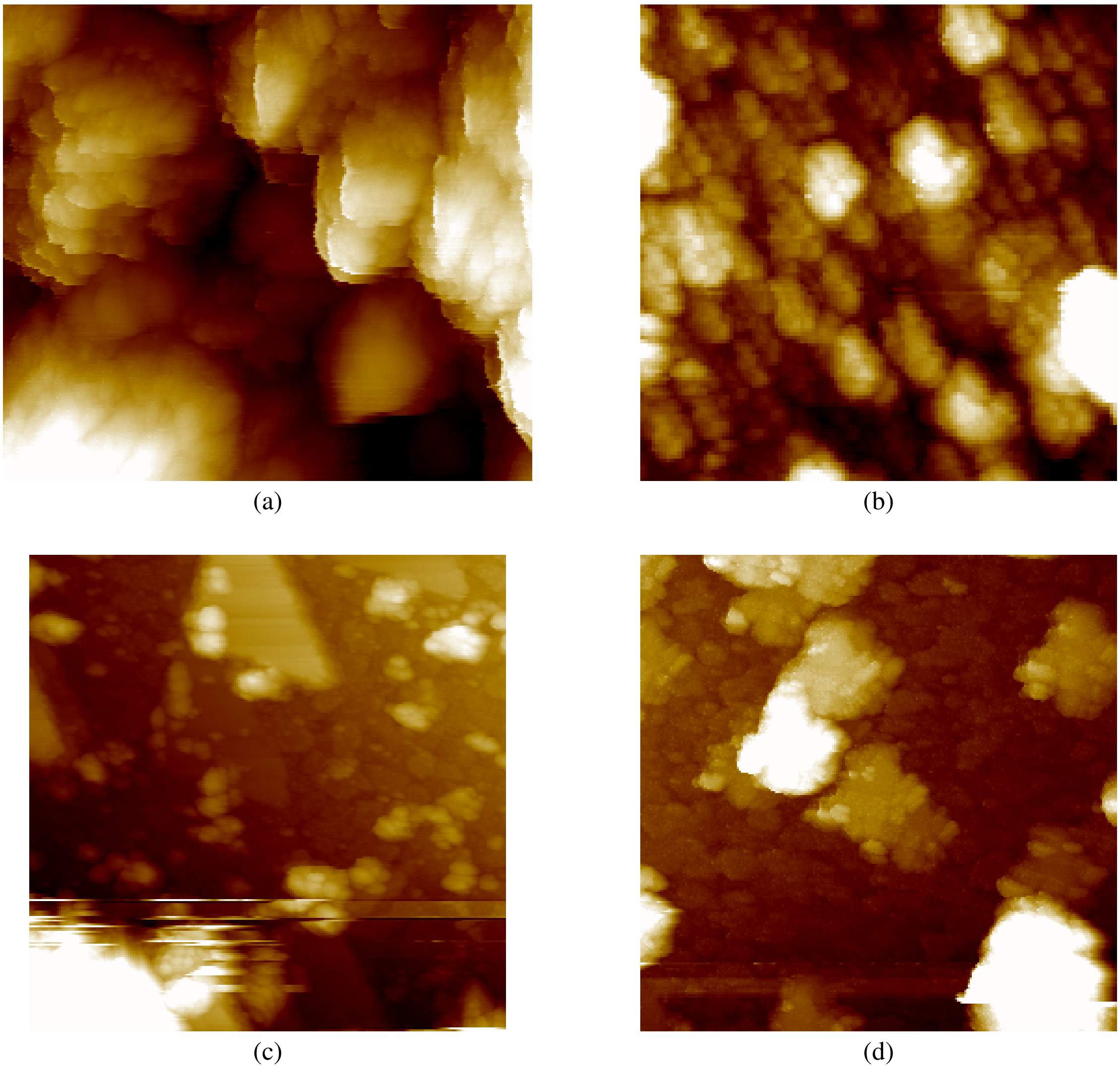

Fig. 1 The AFM topography of TiO_2 thin films by varying the nano-TiO2 powder mass concentrations (a) 1g; (b) 0.4g; (c) 0.1g; (d) 0.05g

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-TiO2 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 TiO2 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 TiO2, 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.

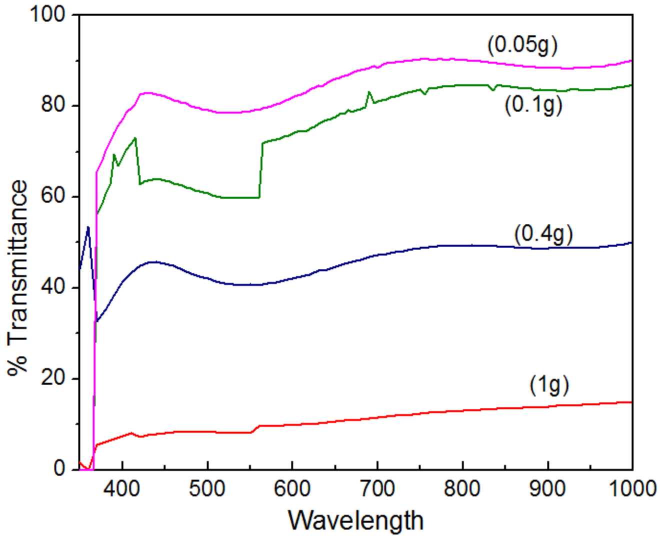

Fig. 2 The transmittance spectra of TiO_2 thin films

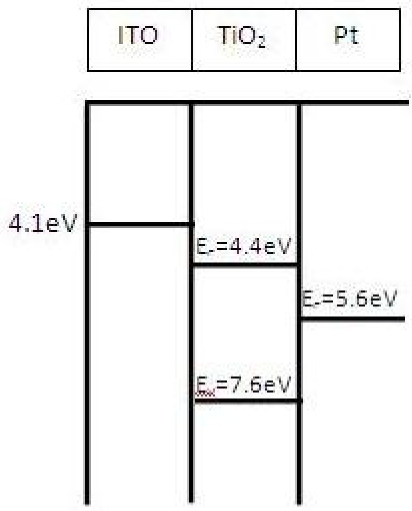

Fig. 3 The band off-set of ITO/TiO_2/Pt contact

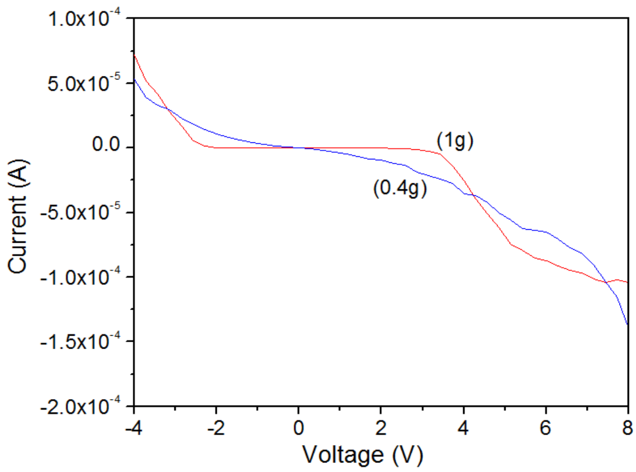

Fig. 4 The Schottky behavior of TiO_2 thin films

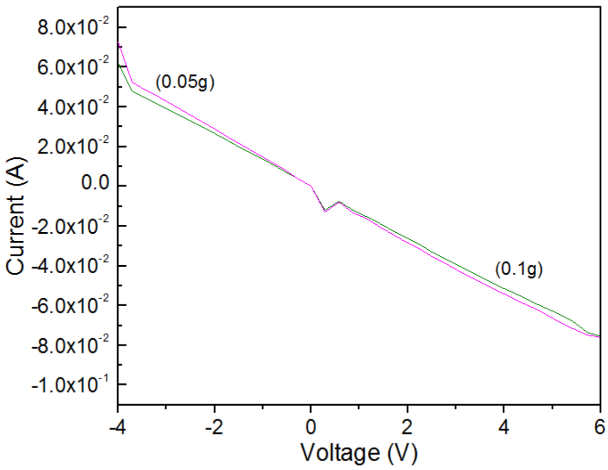

Fig. 5 The Ohmic behavior of TiO_2 thin films

IV. CONCLUSION

As a conclusion, Ilmenite is a good material to fabricate uniform TiO_2 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-TiO_2 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

A special thanks to Mint-SRC, Mechanical Engineering Laboratory and Science Chemistry Laboratory of Universiti Tun Hussein Onn Malaysia for providing the equipment needed.

VI. REFERENCES

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