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STRUCTURAL INVESTIGATION AND PROPERTIES OF TiO_2 THIN FILM PREPARED BY SOL-GEL SPIN COATING

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ABSTRACT

We report the structural properties of TiO_2 (Titanium dioxide) thin films grown using sol-gel spin coating method for temperature treatment (with temperature 50° C and without temperature) The difference in temperature is carried out to determine changes in the surface of the sample by using heating or not. Ideally, the thickness of the TiO2 layer ranges from 10-15 μm, therefore in this study it was carried out in 5 layers. Field Emission Scanning Electron Microscopy (FESEM) consist of surface morphology, Cross-section sample, and EDX images, respectively, providing the structure of the surface. The result confirm that temperature treatment can damage the surface coating, this is proven by the shape of the crack in the coating.
Keywords: DSSC; morphology; spin coating; TiO_2

INTRODUCTION

The career of Swiss scientist Michael Gratzel, who invented dye-sensitive solar cells (DSSCs), marked the beginning of the development of these devices. By using the photovoltaic effect, this sandwich-shaped device's basic operation turns solar energy into electricity [1][2]. As non-renewable energy sources, such fossil fuels, oil, and biomass, are known to be short-lived, DSSCs offer a great deal of potential for the future. The use of DSSCs for renewable energy will therefore be crucial in the future, particularly for solar energy [3].

Solar energy currently comes in three generations, the first of which stands for conventional cells, the second for thin-film cells, and the third for third-generation cells [3][4]. The first and second generation cells are less efficient, expensive fabrication cost, and the unstable absorber materials [3][5]. While currently the issue that is being discussed a lot is how to achieve low cost and highly efficient solar cells [4]. This third generation solar cell will be the main topic in this research, this technology uses organic solar cell materials and polymers

[3]. Dye Sensitized Solar Cells (DSSCs) are third generation solar cells pioneered by Michael Gratzel.

DSSCs consist of four main components including working electrode, dye-sensitized, electrolyte, and counter electrode [6]. Working electrode is the most important part, this is due to on the surface of the working electrode there is a semiconductor layer. The excited electron is injected into semiconductor surface (conduction band) and the electricity is supplied to the counter electrode [6][7][8]. In the process, the solar cell device is an amalgamation of p-n junctions in semiconductor materials, where the p-type semiconductor produces holes in the valence band because it has a number of holes that are more than the number of electrons. When photons with energy equal to or higher than the material's energy band width irradiate a p-n junction, electrons will be excited from the valence band to the conduction band, creating holes in the valence band[6]. The movement of holes and electrons in the material will produce electron-hole pairs and a potential difference will occur when resistance is given to the solar cell terminals. Semiconductors commonly used in the world of research DSSCs are ZnO, TiO_2, SnO, Cu2O [6].

The semiconductor material used in this study is TiO_2 which is an n-type semiconductor which is transparent to visible light and its use was introduced by Gratzel and O'Regan. TiO_2 thin film is the paramount material due to its potentials such as large optical band gap, has a good thermal, and chemically stability [9]. TiO_2 contains three crystal structures that support the reason why TiO_2 was used in this study, including rutile, anatase, and brookite. The highest adsorption is owned by anatase, this is because anatase type TiO_2 has many pores and wide surface area, making it easier to absorb photons [6][10][11].

In addition to the crystal phase, the surface shape of the semiconductor layer has a significant impact, the layers will also aid in enhancing energy harvesting to increase DSSC performance [12] and the authors are aware of the distinctions between surfaces with and without temperature from the research done. Since the surface shape depends on the method and layer used, in this study we will concentrate on coating variations using the spin coating method to obtain the best coating results from semiconductor TiO_2.

METHOD

Preparation of Glass Substrate

The substrate used in this study is conductive glass, namely the type of Fluorine Tin Oxide (Sn:F or FTO) which functions as the body of a solar cell, one of which has electrical properties. The use of FTO glass is due to the need for a sintering process at a temperature of 450^{o} C in research and FTO has a quite strong material and resistant to high temperatures [13-16]. Before the research was conducted, all tools and materials to be used were washed using an ultrasonic cleaner with a solution of aquadest, ethanol, and acetone.

Preparation of TiO_2

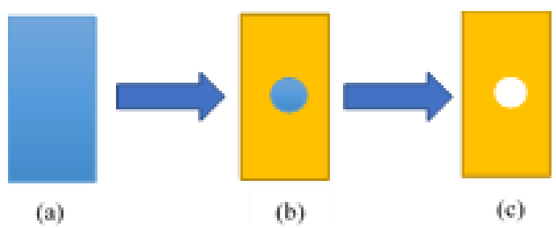

Figure 1. Image of FTO masking area: (a) FTO glass without mask (b) FTO glass with area masking (round shape) (c) FTO glass masking area which deposited by TiO_2

The first step is to make a TiO_2 solution using the following ingredients: 2-propanol, glacial acetic acid, Titanium (IV) Isopropoxide, Triton X-100, deionized water. All the ingredients are then mixed in one container (beaker glass) heat at temperature 50^o C and without temperature, also stirred using a magnetic stirrer for 24 hours.

Next, deposition of the solution on the FTO glass. To determine the conductive side of the glass can be tested using a multimeter. The next stage is to determine the area or masking area on the conductive glass side. The mask used is a tape with the Nitto Tape brand (a special tape that can withstand high sintering temperatures). The masking area on the FTO glass is 0.25 cm^2 as shown in Figure 1.

The method used in this research is spin coating (Figure 2). The solution that has been aging for 24 hours is then dripped onto the side of the conductive glass which has been vacuumed in a spin coater, drop 5 drops of TiO_2 solution for one thin film layer, then spin at a speed of 3000 rpm for 40 seconds. Next, the sample is heated for 1 minute on a hotplate and repeated again from the initial step until the desired number of layers.

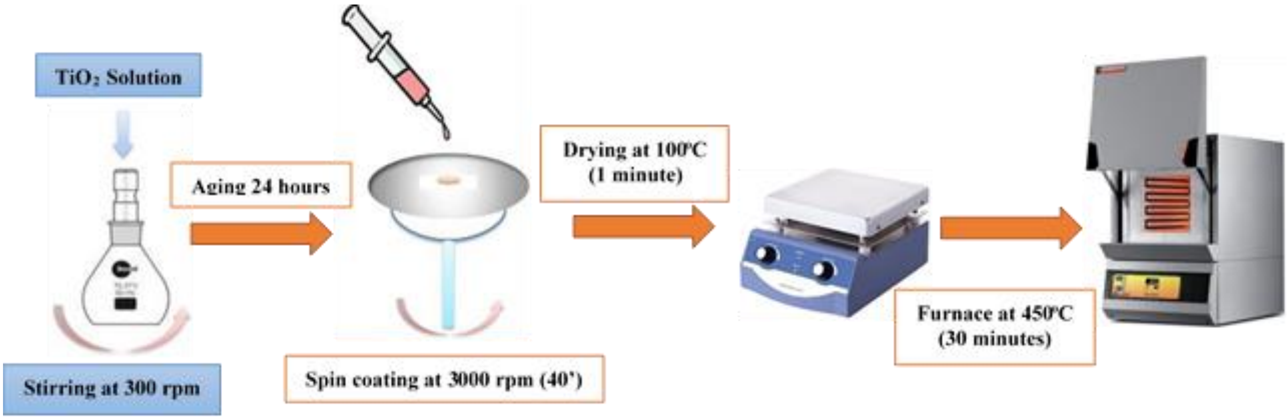

Figure 2. Preparation of TiO_2 thin film using spin coating method

Characterization

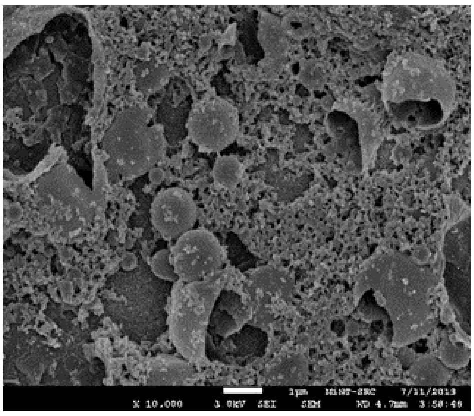

Figure 3. FESEM image of FTO-TiO_2:LEG4-Ag [6]

The characterization used in this research is Field Emission Scanning Electron Microscopy (FESEM), FESEM is an electron microscope that is used to analyze the surface morphology of a material layer in three dimensions by scanning electrons. In FESEM, characterization results can be obtained in the form of surface morphology, cross section, and EDX (Figure 3).

RESULTS AND DISCUSSION

Characterization using FESEM resulted in 3 test samples for each TiO_2 with or without temperature, namely surface morphology, cross section sample, and EDX. After deposition on the FTO conductive glass using a spin coating, the sample was put into a furnace at a temperature of 450^{o}C for 30 minutes. TiO_2 sample used 5 layers (1 layer means 5 drop of solution). The results of the characterization can be seen in the following discussion:

Scanning for Surface Morphology

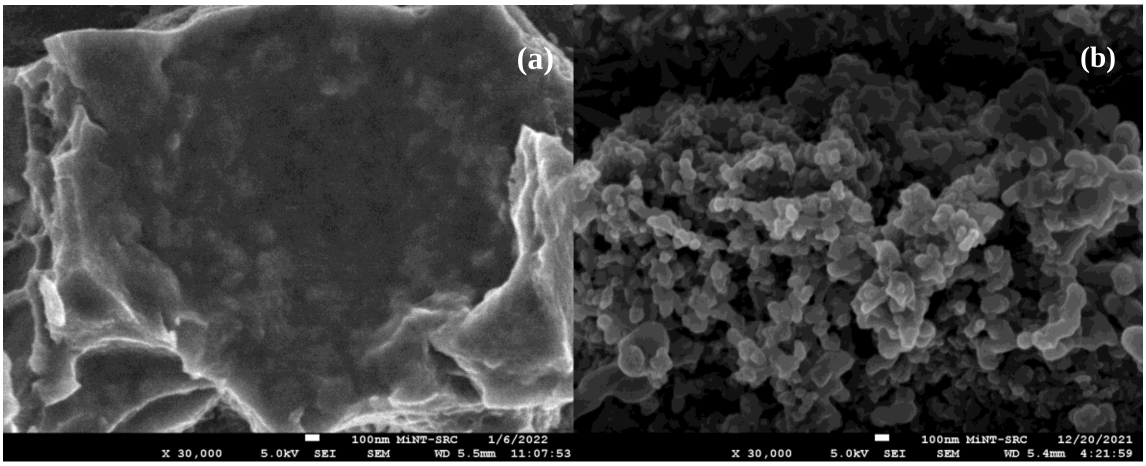

Figure 4. FESEM image of: (a) Surface morphology of TiO_2 with temperature 50^{o}C (b) Surface morphology of TiO_2 with no temperature

The first test was conducted to determine the structure of the surface morphology of the TiO_2 sample coated on the FTO glass layer. Figure 4. shows a significant difference between the two same samples with different temperature treatments. Figure 4(a). is the morphological structure for TiO_2 thin film with temperature treatment (50^{o}C) stirrer for 1 hour. The picture shows that the TiO_2 surface is cracked, so that the surface pores are not visible [17]. The crack that appears is possible because of the temperature treatment during stirrer and when heating on the hotplate after coating the layer, the hotplate temperature used for heating the coat is 100^{o}C for 1 minute. While the small lines around the crack are FTO glass, this means that the particle distribution is not perfectly evenly distributed and the temperature is too high will damage the layer structure.

Figure 4(b). is a sample of TiO_2 without using temperature treatment, with a magnification of 50,000 x it is very clear that the TiO_2 particles are irregularly shaped. From this figure, it can be analyzed that the TiO_2 layer has a porous morphology. This strongly supports the performance of DSSCs, where the surface layer that produces a lot of pores will help in the absorption of dye so that it can maximize its function as harvesting energy [17-18].

Scanning for Cross Section Sample

Figure 5. shows the difference between the two samples (cross section) is clear, where TiO_2 which has a temperature treatment (Figure 5(a)) appears to be cracked. This will make it difficult for electrons to get to the conductive layer, because the shape of the surface is very difficult to reach by light.

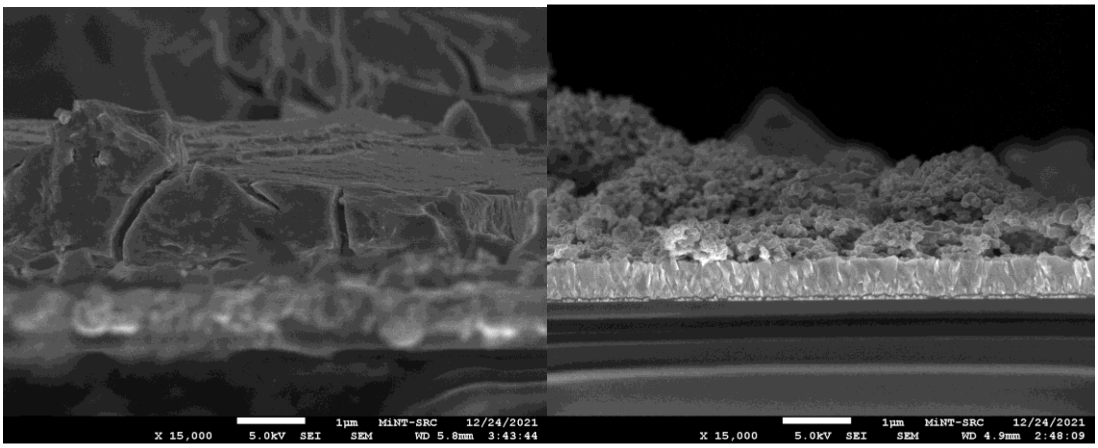

Figure 5. FESEM image of: (a) Cross section sample of TiO_2 with temperature 50°C (b) Cross section sample of TiO_2 with no temperature

Furthermore, it can be clearly seen that the samples that did not use temperature treatment had a nanoparticle structure (Figure 5(b)), but with different heights. The image has a structure that is easily traversed by light, due to the shape of the surface and appears to have many pores so that it will assist in the creation of electron-hole pairs so that it will be able to improve the performance of DSSCs.

Scanning for EDX Sample

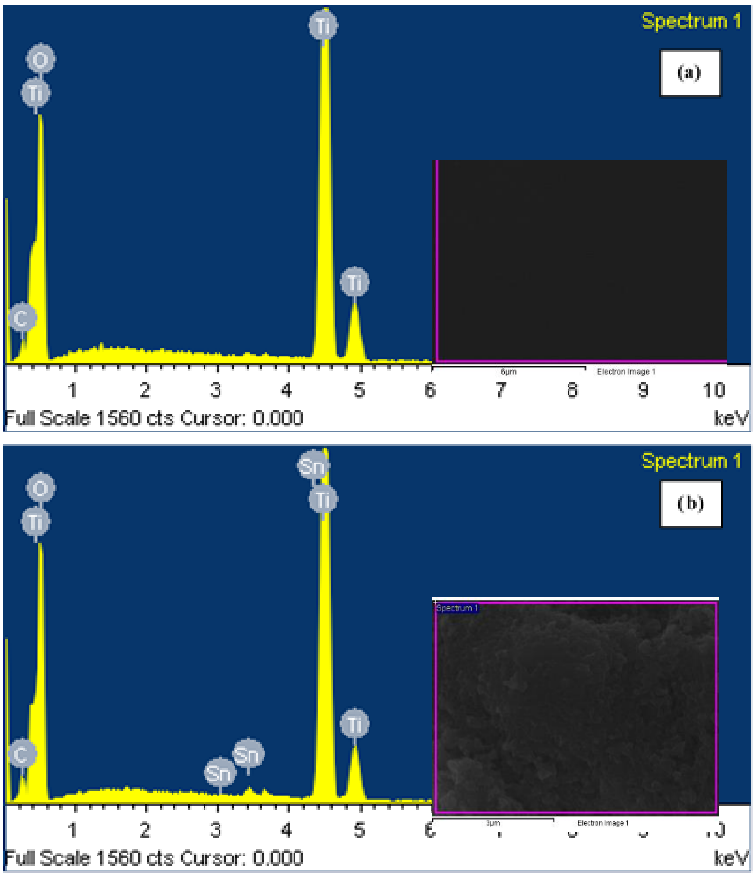

Figure 6. FESEM image of: (a) EDX sample of TiO_2 with temperature 50°C (b) EDX sample of TiO_2 with no temperature

In order to confirm the presence of TiO_2 in the sample we proved it with the EDX analysis (Figure 6(a-b)). It can be observed from the EDX images that we can see the peaks indicating each element of material research that we used. Figure 6(a) shows that ratio Ti, O, and C completely present, but if we compare with Figure 6(b) it found the presence of Sn, O (FTO glass) and also Ti and O. It can be seen on Figure 6(a) that there is no presence of Sn (FTO glass), this is due to the crack of surface because of temperature treatment.

CONCLUSION

In this study, TiO_2 thin film was made by spin coating method and consist of 5 layers which 1 layer is 5 drop solution. In this research using two treatments, including TiO_2 thin film with temperature and without temperature. From FESEM characterization can be known as three samples (surface morphology, cross section, and EDX). These samples show that TiO_2 thin film without temperature treatment has a good surface in the form of nanoparticle. This will facilitate the passage of electrons to the conductive layer and can improve the performance of DSSCs.

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