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The Effect of Water Bath Temperature during Electrochemical Deposition of Zinc Oxide

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Abstract: Nanostructure Zinc Oxide (ZnO) is widely applied in various fields such as optoelectronics, sensors, transducers and biomedical sciences. Nowadays, there are many methods that can be used to produce ZnO thin film such as electrochemical deposition. This method is a process of converting or forming an oxide layer on the metal surface. The purpose of this research is to study the effect of water temperature on the morphology of ZnO during the electrochemical deposition process. ZnO nanostructures obtained after electrochemical deposition process is carried out at different water temperatures and a constant current density. Electrochemical deposition process was carried out when the water temperature reached 60, 65, 70, 75 and 80ºC. During the deposition process, 0.05 M/L zinc nitrate is used as a single electrolyte. The analysis conducted is divided into four categories, namely FESEM, XRD, EDX and UV-Vis. As a result, the suitable water bath temperature for forming the ZnO morphology with an electric current density of 4 mA/cm^2 is at 75ºC and at this temperature it has a higher value of composition mass where is 0.61 % compare with other temperature. From the UV-Vis analysis, it shows the value of band gap is 2.87 eV when the temperature of water bath was 75ºC. Various studies were conducted to produce ZnO nanotubes because it provide different forms of reaction temperature at constant current densities.

Keyword: Zinc oxide; Electrochemical deposition; XRD; FESEM; UV-vis

1. Introduction

Electrochemical deposition is a process that involves the reduction of metal ions from aqueous, organic and fused salt electrolyte [1]. Based on the experience in industrial history, the deposition is developed for metal plating process. The researchers found that this method can provide vast opportunities especially in areas related to the synthesis of metal oxides, alloys and materials that have nanostructures. Basically, the process involves electrochemical deposition of heterogeneous electron transfer between the electrode and the electroactive material contained in the liquid in which the change in the nature of crystalline metal atom [2].

Mr. Green has introduced the use of nanomaterials in the design of the solar cell layer main aims of the 3rd generation is to produce a layer of solar cells on low production costs but have a high level of energy efficiency [3]. Electrochemical deposition or known as electrochemical plating process is quicker and cheaper compare with the other production techniques of oxide material which is mainly involve fabricating the semiconductor industry and energy storage material. This method is also frequently used by manufacturers of canned foods to be used as corrosion protection.

In recent years, researchers have shown interest in relation to the production of metal oxide thin films by electrochemical deposition (ECD) methods. Zinc oxide (ZnO) is an example of a material that has the potential to be used in the production of products such as solar cells, the presence of gas sensors, and transducers supersonic devices. Precipitation electrochemical very different from other methods due to the thickness and surface morphology of ZnO can be controlled by changing certain parameters, deposition rate is high, the cost of the experimental low-

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temperature applications are few and easy to identify foreign objects are present in the sample [4].

The ECD process has been studied at different temperature recently. An increase of the transmittance for the films grown at lower temperatures has been found [5]. In this paper, a study is carried on the morphological dependence on bath temperature during deposition.

2. Experimental Method

The ZnO nanostructure was deposited by ECD technique through $Zn(NO_3)_2$ aqueous solutions at room temperature. Using a simple two electrode, a flat plate FTO was used as the anode and the Silver substrate as the cathode, as shown in Fig. 1. The distance between the electrodes was about 0.5 cm. Electrodeposition was carried out at constant current density of 4 mA/cm^2. The only parameter of water bath temperature was changed in the experiment are range at 60, 65, 70, 75 and 80 ^oC. The deposition time was 30 minutes while all the products of ZnO film were then annealed at 260 ^oC for 2 hours under oxygen ambient.

The crystal phases of the ZnO were studied by x-ray diffraction (XRD) using the Bruker model D8 Advance, which was operated at 60 kV. The morphologies of the ZnO were characterized by a field emission scanning electron microscope (FESEM) JSM-7600F JEOL with an accelerating voltage of 10.0 kV. To obtain the thickness of the formed ZnO layer, surface profilometer KLA Tencor Alpha Step IQ was used. The optical properties of the ZnO were analyse by using UV-vis spectrometer.

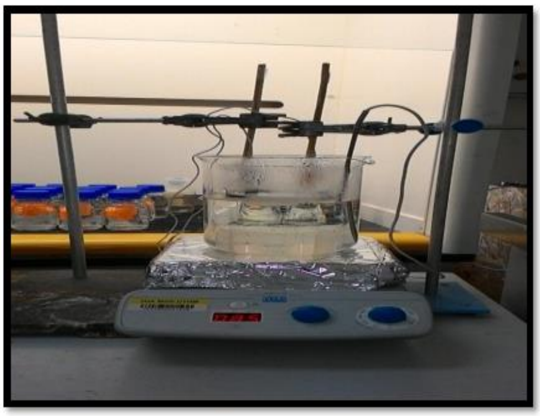

Fig. 1 ZnO electrodeposition apparatus setup

3. Results And Discussion

XRD observation of ZnO. As shown in Fig. 2, the detected (h k l) peaks in agreement with the standard JCPDS 036–1451 card for hexagonal wurtzite ZnO. The existence of ZnO and Sn visible on the surface of the substrate. Sn shows higher peak compare with the peak of ZnO. This happens because the used of FTO as substrate while the existence of ZnO is in very small amounts that affected by the total molarity of zinc used during the deposition process.

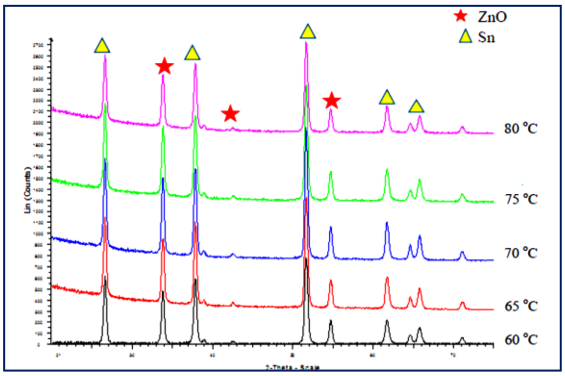

Fig. 2 XRD pattern of 2θ for each sample with different temperature of water bath.

Through the analysis, the highest intensity of ZnO is at (101) plane with water bath temperature of 75ºC. The higher intensity of (101) plane indicates that the morphology of ZnO nanostructure is clearly the best when the deposition water bath temperature is 75ºC. This result is in good consistent with the SEM micrograph that will be discussed in the next section. The formation of the ZnO nanostructure for all samples has similarities with the studies conducted by Mutukumar *et al.*, (2013), where ZnO successfully forms on (101) plane at 2θ of 36º [6].

Energy dispersion X-ray (EDX) is performed to determine the presence of essential elements found on the surface of the substrate. EDX analysis is very important in this study because it proved the presence of the elements zinc and oxygen to be produced ZnO. In addition, EDX can support XRD analysis in the process of producing ZnO samples.

The increasing of composition of ZnO can be observed when the water bath temperature increase from 60ºC to 75ºC. At water bath temperature of 80ºC, a decrease in composition percentage of ZnO occurs. This results clearly consistent with the XRD results that shows high intensity of (101) plane transpire at 75°C water bath temperature.

Morphology of ZnO films: Fig. 3 shows the morphologies of ZnO nanostructure after electrochemical deposition process at a 4 mA/cm^2 of constant current density and at different water bath temperature. ZnO structure morphologies are taken by using FESEM with magnification range between x50,000 to x100,000. The growth of ZnO nanostructure began to form at the water bath temperature of 65°C where the particles are visible overlap on each other. ZnO nanostructure rod-shaped formed when the water bath temperature was increased to 75°C.

The formation of ZnO particles occurred due to ion Zn^{2+} separate from the influence of an electric field to the cell. When the water bath temperature is increased, ion Zn^{2+} move faster and subsequently dissolved in water. This situation enables the plating of ZnO particles on the substrate. The formation of ZnO occurs when ions Zn^{2+} forms a bond with oxygen. The composition of ZnO structure at 75°C water bath temperature seem more compact than the other samples.

FESEM images showed inconsistencies nanostructure ZnO occurs when the water bath temperature is changed to 80°C. The increase in water bath temperature caused the formation of ZnO particles become more unstable and have various shapes [5].

ZnO nanostructures on a layer FTO influenced by the temperature of the water bath and the current density that be used. The type of nanostructures formed after electrochemical deposition treatment by different water bath temperature resulted by coalescence of neighbouring nanorods due to Ostwald ripening. Ostwald ripening is one of phenomenon in which smaller particle in solution dissolved and deposited in the large particle in order to achieve more solid basis of thermodynamically state, where the surface ratio is reduced [7].

UV-Vis absorption spectra analysis: Ultra-violet Spectroscopy (UV-Vis) was used to measure the optical properties of the ZnO thin films. Fig. 4 shows the graph for absorbance based on the temperatures. The results of absorbance that achieved has much influenced by the condition such as the thin film coating are even or not, surface roughness and impurities that may be contained in the layer are analyzed.

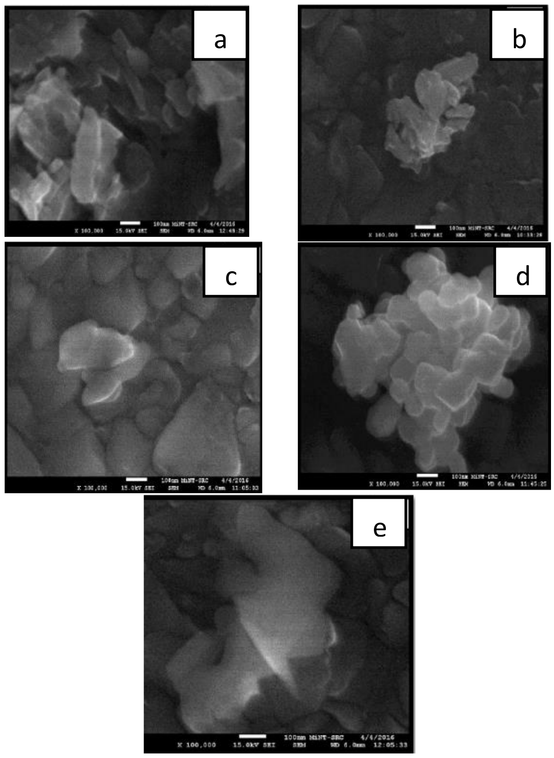

Fig. 3 FESEM image of (a) 60, (b) 65, (c) 70, (d) 75 and (e) 80°C water bath temperature.

The highest absorbance achieved was approximately 0.112% for water bath temperature during deposition of 60°C. The light absorption decrease with the increasing of water bath temperature. According to a study produced by P.H. Vajargah, the graph is a relatively common form in which the temperature is not influenced the percentage of absorbance where the lower temperature getting higher percentage absorbance [8].

The band gap energy of synthesized nanostructures plays an important role in deciding the photocatalytic activity of photocatalyst. The UV–vis absorption spectra of different water bath temperature were studied to calculate the band gap value. Tauc relation was used in order to find the value of direct band gap [9].

$$\alpha h\nu = A(h\nu - E_g)^n \qquad (1)$$

The results shows that the direct band gap values ranging from 2.86 - 3.05 eV. The highest band gap value achieved is 3.05 eV where the water bath temperature is at 75 °C during deposition.

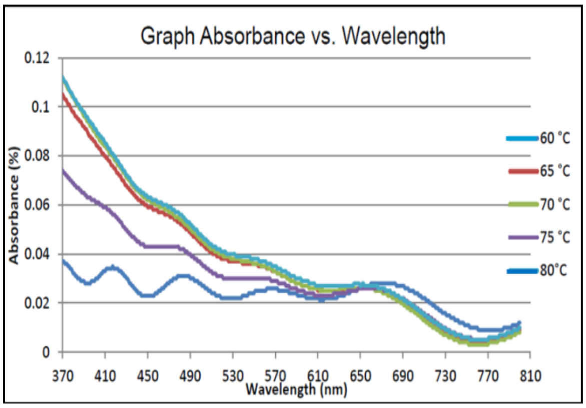

Fig. 4 UV-Vis absorbance vs wavelength at different water bath temperature

4. Conclusion

Generally, the formation of ZnO thin films have been successfully produced by electrochemical deposition process. Each sample ZnO have differences in morphology, size, and thickness depending on the water bath temperature used during deposition. Analysis on morphology and also the nature of the ZnO are generated by using FESEM, EDX, XRD and UV-Vis. Based on the results obtained, the water bath temperature influenced the formation of ZnO nanostructures. However, ZnO particles are successful formed at each sample during deposition. The use of electrochemical anodization process is a challenge for the new generation of semiconductor materials to develop widely.

Acknowledgement

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