Synthesis of Zinc Oxide Nanostructure by Chemical Bath Deposition (CBD) Method: Influence of Growth Time towards Nanostructure Characteristics

R. A. Rahman, M. A. Zulkefle, S. H. Herman and R. I. Alip

Abstract*: Growth of zinc oxide (ZnO) nanostructure on seeded indium tin oxide (ITO) via chemical bath deposition were presented in this study. Growth time is believed to have vital role in order to control the physical (morphology), optical and structural characteristics of ZnO nanostructures. Several growth time of ZnO nanostructure were varied (1 H – 3.5 H) as the purpose to investigate its effect towards the growth of ZnO nanostructures, as well as their characteristics. In this study, the influence of growth time was determined using field emission scanning electron microscope (FESEM), ultra-violet visible spectrometer (UV-Vis) and x-ray diffraction (XRD). Based on the results obtained, morphological, optical and structural characteristics of ZnO nanostructure thin films grown at various growth time present different characteristics and properties. According to the results obtained, it is proved that growth time is a vital parameter to control the ZnO nanostructure growing process. ZnO nanostructure morphological changes significantly with the changes of the growing time process. As well as optical properties, the changes of absorbance and transmittance value influence the optical energy band gap of ZnO nanostructure in this study, which is the average value is 3.31 -3.40 eV. The structural characteristic of the ZnO nanostructure also affected significantly with the difference of growth time, where the crystallinity is improved with the longer growth time.

***Keywords* : chemical bath deposition, crystallinity, growth time, morphological**

I. INTRODUCTION

Nano- semiconductor materials have gained many attention and attraction in these recent years, due to their outstanding properties and capable to be applied in many applications such as photovoltaic, photoelectron devices, photocatalyst, as well as sensors [1-4]. Nanomaterials have few distinguish structural, electronic, thermal and optical properties which are important and impressive to be practiced in either basic or applied fields. Numerous nano-semiconductor materials known to have attraction among researchers such as carbon nanotube (CNT), titanium dioxide nanotube (TiO_2 nanotube), nickel oxide (NiO), as well as zinc oxide (ZnO) nanostructures [5-8]. Among these numerous materials, ZnO is a diverse functional material due to its own properties and functionalities. These properties include the wide band gap of ZnO (3.37 eV) with high exciton binding energy (60meV), biocompatibility, piezoelectric characteristics, as well as easy to be fabricated. Due to these distinguished properties, ZnO is widely applied in many potential applications and fields such as solar cell, light emitting diodes, ultra-violet sensor (UV sensor), optical and electrical devices, as well as sensors (chemical, bio and gas) [9-11].

In addition, ZnO is also one of the materials that possess a numerous family of nanostructures. Nanostructures is predicted (in general) to plays a crucial role in the future, hence this nanostructure can be practices in stated applications earlier thus the performance could be increased. Due to this, a thorough understanding of the growth mechanism needs to be practices in order to accomplish desired nanostructure to suit the application chosen. Since the characteristics of ZnO nanostructure is greatly dependent on its morphological, it is also important to exactly control their shape, surface morphology, and size in order to utilize its properties to be practiced in many applications.

According to the previous researches, there were assorted methods was used and applied to synthesis and produced the nanostructures of ZnO which include electrochemical deposition technique, sputtering method, metal organic chemical vapor deposition (MOCVD), also pulse laser deposition [12-14]. However, these methods require severe reaction condition, for example accurate gas condition, high temperature, suitable flow rate which include the complexity during the process. To divert from these complex methods, a simple method with low temperature can be a good approach [15].

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* Correspondence Author

R. A. Rahman, Integrated Sensor Research Group, Faculty of Electrical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor Malaysia. Email: rohanieza.abdrahman@gmail.com

M. A. Zulkefle, Integrated Sensor Research Group, Faculty of Electrical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor Malaysia. Email: alhadizulkefle@gmail.com

S. H. Herman*, , Integrated Sensor Research Group, Faculty of Electrical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor Malaysia. Email: hana1617@uitm.edu.my

R. I. Alip, , Integrated Sensor Research Group, Faculty of Electrical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor Malaysia. Email: rosalena@uitm.edu.my

Chemical bath deposition (CBD) can be an alternative method to produce and ssythesis ZnO nanostructure since this method does not need flow rate application with the usage of low temperature, thus the growth parameters could be controlled effortlessly. Simple setup, low cost, large area deposition, and eco-friendly are also part of CBD method advantages.

The growth of ZnO nanostructures could be affected by several parameters such as seeding of the substrate (the growth of ZnO nanostructures will improve with seed layer), thickness or layers of seed layer, position of substrate during growth process, temperature, pH, concentration and also growth time. It is believed that these parameters will influence the shape, size, length as well as the diameter of the nanostructures.

In this study, CBD method was used to synthesis ZnO nanostructures. Growth time parameters were explored in to examine the characteristics and properties of the ZnO nanostructure. The results demonstrate that ZnO nanostructures shows different morphology, structural and optical characteristics, hence prove that this parameter influence the growth and characteristics of ZnO nanostructures.

II. EXPERIMENTAL

There were three different steps involve in this study, which include the preparation of seed layer (ZnO seed layer), ZnO nanostructure growth process and characterization process. Before the ITO substrate was deposited with ZnO seed layer, this substrate was first cleaned using standard cleaning process. All of the substrates were immersed in methanol (C_2H_5OH) and were sonicated for 10 minutes at 50 °C. After that, C_2H_5OH was discarded, then replaced with deionized (DI) water. The same sonicating conditions were applied. Then, DI water was also discarded. Before been blown with argon (Ar) gas, the substrate was rinsed few times to ensure that all of the contaminations were eliminated. Lastly, cleaned substrates were kept in small container to keep from contamination before been deposited with ZnO seed layer.

A. ZnO Seed Layer Deposition

Seed layer of 0.4M ZnO solution was synthesized with the usage of zinc acetate dihydrate ($Zn(O_2CCH_3)_2$), monoethanolamine (MEA, $HOCH_2CH_2NH_2$), and 2-methoxyethanol ($C_3H_8O_2$). Zinc acetate act as the precursor, MEA as the stabilizer while 2-methoxyethanol is the solvent for the solution. These chemicals then were mixed, as well as stirred together at 300 rpm. For the first 3 hours, 80 °C heat was applied. After 3 hours, the heat was switched off and the solution was kept stirred for another 24 hours to obtain a clear solution. After aging process, prepared ZnO solution was deposited as the seed layer onto substrate via spin coating technique. 10 drops of ZnO solutions were spun at 3000 rpm onto the substrate for 60 seconds, then this seed layer was dried at 150 °C for 10 minutes. After drying process, the annealing process took part where the prepared seed layer was annealed for 1 hour at 500°C.

B. Growth of ZnO Nanostructure using CBD Method

The aqueous solution for the growth of ZnO nanostructures was prepared using zinc nitrate ($Zn(NO_3)_2 \cdot 6H_2O$, 99%) and hexamethylenetetramine (HMT) ($C_6H_{12}N_4$, 99.5%), with the molar ratio of 1:1 in 500 mL of total solution. This solution was sonicated for 30 minutes at 50 °C before the aging process was continued for 3 hours (stir at 300 rpm). Then, the prepared solution was poured out into different 100 mL schott bottles, before the seeded substrates were inserted. This schott bottles were immersed in water bath, which already setup to 95 °C temperature. The growth process was varied in the range of 1 H, 1.5 H, 2 H, 2.5 H, 3 H and 3.5 H. After growth process, the thin film with ZnO nanostructures were first rinsed with DI water before been dried at 150 °C for 10 minutes. Lastly, the thin films were annealed at 500 °C for 1 hour. The mechanism of growth formation of ZnO nanostructure based of CBD process can be summarized as below [16]:

$$C_6H_{12}N_4 + 6H_2O \leftrightarrow 6HCHO + 4NH_3 \quad (1)$$

$$NH_3 + H_2O \leftrightarrow NH^{4+} + OH^- \quad (2)$$

$$Zn(NO_3)_2 \rightarrow Zn^{2+} + 2NO^{3-} \quad (3)$$

$$Zn^{2+} + 2OH^- \leftrightarrow Zn(OH)_2 \quad (4)$$

$$Zn(OH)^2 \rightarrow ZnO + H_2O \quad (5)$$

C. Characterization Process

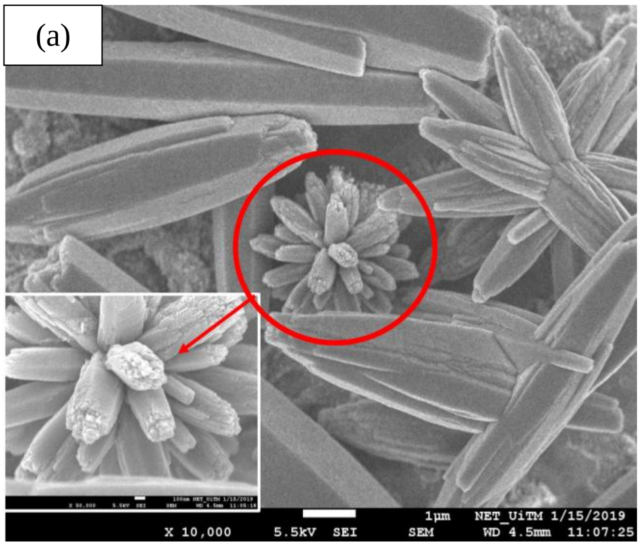
After all of the thin films with ZnO nanostructures were prepared, the characterization process was conducted. Morphological of the ZnO nanostructures were examined using field emission scanning electron microscope (FESEM), while the structural properties of the ZnO nanostructures was confirmed with x-ray diffraction (XRD), then the optical properties of the ZnO nanostructures films were determined using ultra-violet visible spectra (UV-Vis). All of the obtained results were presents in the results and discussion section.

III. RESULT AND DISCUSSION

Field emission scanning electron microscope (FESEM) revealed the morphology structure of the ZnO structure grown at different growth duration. Fig 1 (a) and (b) illustrate the different morphology of ZnO nanostructure grown at 1.0 H and 3.0 H. 1.0 H represent the lowest growth time, while 3.5 H is the highest duration of growth time. These two figures show different morphology, which can be observed that in Fig. 1 (a), ZnO nanostructure flower-shape was formed. However, as the inset figure with 50K magnification, this flower actually is a formation of nanorod, which the rod is not fully formed. This might reasoning to the insufficient growth time, which the growth time need to be increased, so that the ZnO nanorod could form completely.

For ZnO nanostructure in Fig. 1 (b), ununiform ZnO nanorod could be see with the increasing of growth time (3.5 H). By observing in the inset figure, the ZnO nanorod with small size is formed.

Comparing the length of nanorod in both figures, growing process for 3.5 H produces longer and fine nanorod. This result reveal that the different duration of growing process will expose the ZnO nanostructure grown in same solution experienced different growth rate of nanorods. The growth rate of nanostructure could be defined as along with growing length per growth time [17]. Hence, as the time inclined, the nuclei of ZnO will grow continuously. ZnO on the surface of the ZnO seeded substrate is further oxidized from the oxygen to form ZnO nanorods on the substrate [18].



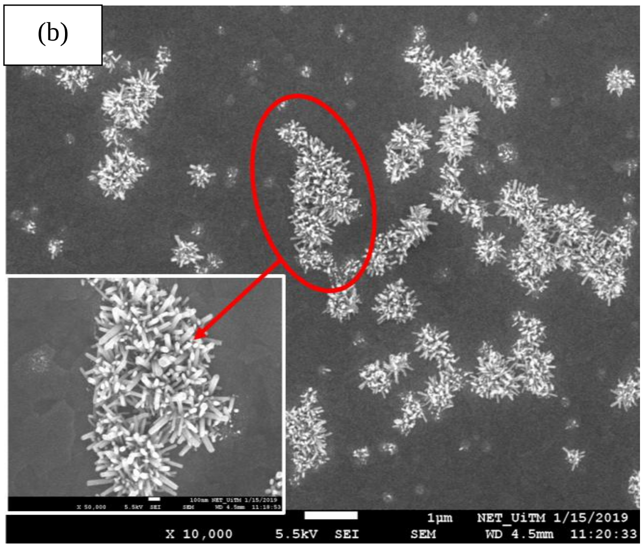


Fig. 1. FESEM images for (a) 1.0 H and (b) 3.5 H growth time at 10K and 50K magnification.

The optical properties of ZnO nanostructure grown on ITO substrate seeded with 0.4M ZnO at different growth time were investigate using ultra-violet visible spectrometer (UV-Vis). The investigation is based on their optical absorption and transmission which the wavelength is ranging from 350 – 800 nm. ITO/glass substrate was used before each of the measurement, as the reference, in order to substrate the effect of the indium tin oxide and the glass. Fig. 2 shows the absorbance measurements as a function of wavelength for ZnO nanostructures grown on different growth time (1-3.5 hours). According to the graph obtained, ZnO nanostructure exhibited higher absorbance values at short wavelength (below than 400 nm), which reaching the lowest values at the IR region. This absorption characteristics is typically belongs to the ZnO characteristics, which stated by L. Roza et al. in their study. [19]. In addition, the absorption edges are below 400 nm also attribute to the intrinsic ZnO band gap because of electron transitions from the valence band to the conduction band.

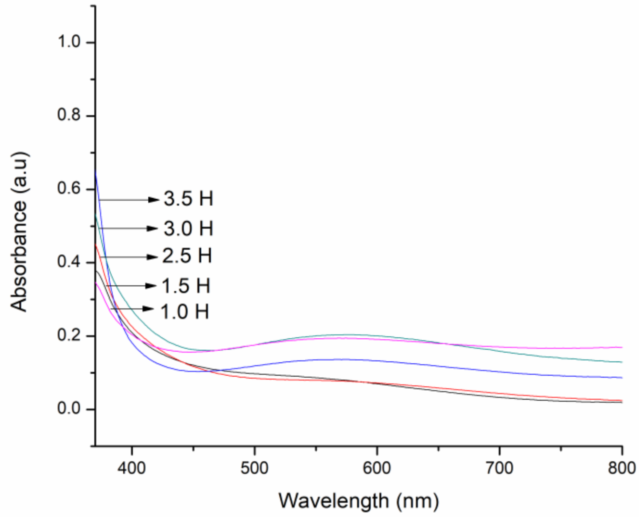


Fig. 2. Absorbance spectra for ZnO nanostructure grown at different growth time

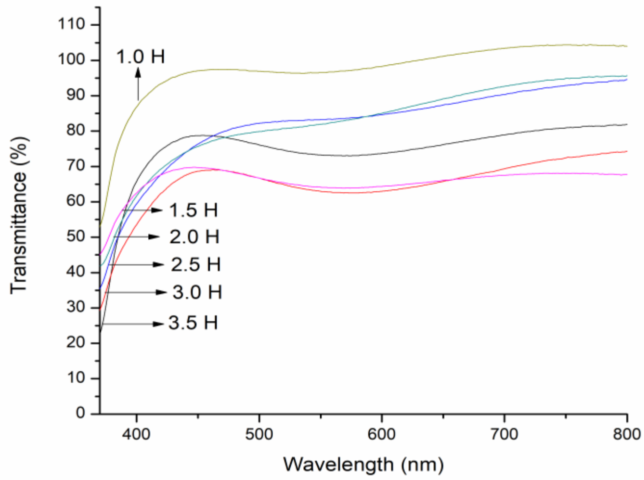


Fig. 3. The optical transmittance of ZnO nanostructure with different growth time

As can be observed in the results obtained, the absorbance of the ZnO nanostructure grown at different growth time increased with the increasing growth duration. This finding is also supported by the finding reported by A. F. Abdul Rahman et al. and A. Yengantiwar et al., where they also gained the same trend of absorbance when the growth time was increased [20][21]. This phenomenon might be due to the increasing of hydroxide accumulation along grain boundaries and topological changes of the ZnO nanostructures when the growth time increase [22]. As for the transmittance, the results obtained could be observed in Fig.3 with the wavelength of 370-800 nm.

It can be observed that all of the ZnO nanostructure grown at different growth time present high transmittance value in the UV region. The transmittance spectra revealed that ZnO nanostructures had the average transmittance value of 45.8%. Highest transmittance is found for ZnO nanostructure grown at 1.0 H, which is 54.7%, while the lowest transmittance value belongs to ZnO nanostructures grown at longest duration (3.5H), with the percentage of 24.8%. According to the data obtained, the transmittance value for all of the grown ZnO nanostructure decrease when the growth time is increased. The decreasing of the transmittance value might attribute from the scattering effect that has been enhanced during longer growing process [23]. Other than that, the reducing of the transmittance value also due to the few factors which include the optical scattering at the grain boundaries, c-axis orientation, as well as the film thickness [24]. When the ZnO is grown at longer time and duration, the thickness of the film might affect, where the film will become thicker, as found by M. F. Malek et al. in their study [25]. This increase of the thickness however will influence the optical scattering which reduce the transmittance of the ZnO nanostructures thin film [24].

The optical band gap of ZnO nanostructures grown at different growth time is calculated using the formula stated below:

$$Eg = \frac{hc}{\lambda} \tag{6}$$

Where,

Eg	=	optical band gap energy
h	=	Planck's constant
c	=	the speed of light in vacuum
λ	=	wavelength

Based on the calculation, the optical energy band gap for all of the ZnO nanostructures grown at different time gave the slightly different in value between each other. The optical band gap obtained are ranging from 3.31 – 3.40 eV. All of the optical band gap energy attained agrees well with the theoretical band gap value, which is 3.37 eV. Generally, the optical energy band gap of ZnO nanorods are lower rather than ZnO bulk and about 3.37 eV. However, in this study, the value is slightly different due to the optical confinement effect of the formation of ZnO nanostructure [26].

Meanwhile, X-ray diffraction (XRD) was used to examine the crystallinity of the ZnO nanostructure grown at different duration. Fig. 4 presents the XRD spectra for ZnO nanostructure synthesis at different time. From the XRD pattern, it can be observed that the major and dominant peak of ZnO occurs at 2θ = 34.49 that corresponding to (100) orientation. This major peak could be detected during the growth time of 2.5 H. However, during another growth time, (100) orientation also could be detected, which is minor peak can be seen at 2θ = 32.49, 32.33, 30.65, and 30.89. Besides, (002) orientation with minor peak also can be observed at 2θ = 35.66, 35.8, and 35.85. For ZnO nanostructure grown at 2.5 H, a minor peak of (112) orientation is detected, which belongs to 2θ = 66.84. As reported by A. B. Rosli, (100) peak is correlated to the lateral growth of ZnO [27].

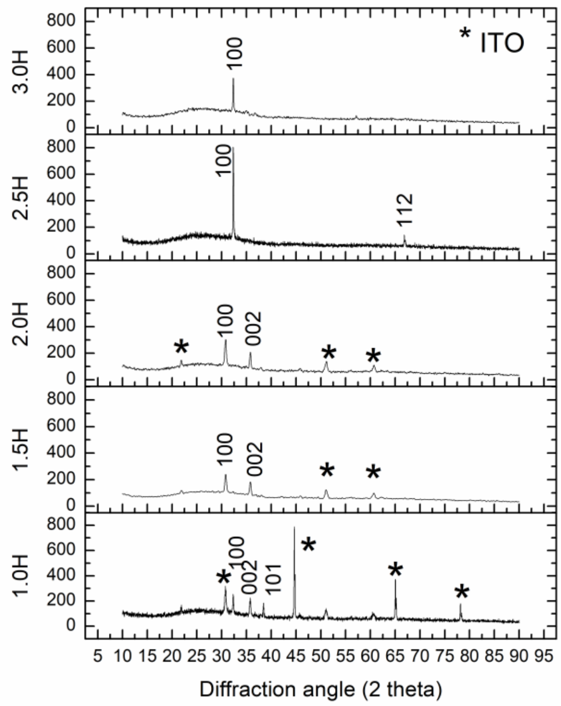


Fig. 4. XRD pattern for ZnO nanostructure grown at 1.0 H, 1.5 H, 2.0 H, 2.5 H and 3.0 H.

According to the results, the peak intensity of (100) orientation is increased when the growth time is increased. This trend can be observed from ZnO nanostructure grown at 1.0 H until 2.5 H, where the peak intensity become higher with the increase of synthesis time. This phenomenon indicates that ZnO nanorods growth along c-axis with excellent crystallinity. However, this peak intensity decreased when further growth time is increased (3.0 H). This finding is an agreement with the finding reported by L. M. Fudzi et al, which they also gain the same trend of XRD pattern when the reaction time of ZnO nanostructure is increased [28].

IV. CONCLUSION

ZnO nanostructure has been successfully synthesized onto the ZnO seeded substrate by chemical bath deposition in this study. The influence of the growth time on morphological, optical properties (absorbance, transmittance and optical energy badn gap) and structural properties are studied. It was found that the growth time affect the formation of ZnO nanorod, where longer growth time is needed to form an uniform structure. In addition, growth time is also essential in controlling the shape, size, distribution and alignment of ZnO nanorod. The increasing absorbance and decreasing transmittance are also influenced by the growing duration, thus this give the effect towards optical energy band gap (3.31-3.40 eV). However, the values of the optical energy band gap is still approaching the theoretical band gap of ZnO which is 3.37 eV. Lastly, the crystallinity of the ZnO nanostructure is also confirmed using XRD, which the results shows that the increase of growth time increases the intensity of (100) orientation.

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AUTHORS PROFILE

Rohanieza Abdul Rahman is a Bachelor in Science (Hons) Chemistry holder, from Universiti Teknologi MARA (UiTM) in 2013. She received her Master in Science (Electrical Engineering) from also Universiti Teknologi MARA (UiTM) in 2018. During her master's study, her research interest is focusing on bilayer thin film (TiO_2/ZnO), which this thin film was applied as pH sensor. Currently, she is pursuing her Ph. D degree in Electrical Engineering at Universiti Teknologi MARA (UiTM). Her research is focusing on fabrication of ZnO nanostructure/P3HT/CuO and solar cell application. Rohanieza published few articles and conference proceeding regarding her research interest during her master's study period.

Muhammad AlHadi Zulkefle received his Bachelor of Science (Material Technology) with Honours (2014) degrees from Universiti Teknologi MARA (UiTM) Shah Alam, Malaysia. He also received his M. Sc. Degree in Electrical Engineering from Universiti Teknologi MARA (UiTM) in 2018. His master's study was focusing on fabricating and characterizing TiO_2 thin film that could be applied as pH sensors. He published few articles and conference proceeding on TiO_2 thin film as pH sensors. Coming after, he is undergoing his Ph.D in Electrical Engineering, which also focusing on the application of pH sensor. However, his research interest in metal oxide is expanded to new material, which is Ta_2O_5.

Sukreen Hana Herman received her Bachelor of Engineering (Electrical, Electronics and Information) from Kanazawa University, Japan in 1999 and Master of Engineering (Electronics and Computer Science) from Kanazawa University (2004). She received her PhD in Materials Science and Technology in 2009 from Japan Advance Institute of Science and Technology (JAIST). During her study, she received the Malaysian Public Service Department (JPA) Scholarship (degree), Panasonic Scholarship (Master) and Japan Monbukagakusho (MEXT)(Ph.D).

Sukreen Hana is currently a senior lecturer at Faculty of Electrical Engineering, Universiti Teknologi MARA (UiTM). She started to join UiTM since 2004, after graduated from JAIST. Besides, she is also the head of Integrated Sensor Research Group (DERIA). Her research interest is including fabrication and characterization of metal oxides, nanostructure materials, and sensors. She received various awards in invention and innovation exhibition and competition. In addition, she is also one of the authors that have more than 170 articles and conference proceedings.

Rosalena Irma Alip received both of her bachelor's degree in Electronics Engineering and master's degree in Nano-material System Engineering from Gunma University, Japan. She also received her Ph.D in Advanced Production System Engineering from the same university. During her Ph.D, she had discover a novel structure foa non-volatile memory that uses phase-change mechanism to store memory.

Rosalena Irma currently is a senior lecturer at Faculty of Electrical Engineering, University Teknologi MARA (UiTM). Also, she has working experience in various field, such as biosensor, humidity sensors, non-volatile memory and solar cell. In Current research, she is focusing on phase change memory, phase change materials and solar cell.