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Zno/Ag Thin Layer Microstructure with The Effect of Annealing Temperature

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Abstracts

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Received: 01 November 2018,
Revised : 20 November 2018
Accepted: 01 Desember 2018

The formation of ZnO/Ag morphology in the form of ganglia structures that are overgrown with grains in previous studies has been shown to degrade the presence of E. coli bacteria. In this research, the variations of annealing temperature were studied, namely 250 °C, 300 °C, 350 °C, and 400 °C when the 4% ZnO/Ag deposition had an effect on crystallinity and morphology. The crystallinity of ZnO/Ag was obtained by using the X-Ray Diffraction (XRD) test and the surface morphology of the ZnO/Ag layer using the Scanning Electron Microscope (SEM) test. The results of the research with the XRD test showed that the crystal structure of ZnO/Ag 4% was hexagonal wurtzite at annealing temperature of 250 °C and 300 °C, while the amorphous structure was obtained in ZnO/Ag with annealing temperature of 350 °C and 400 °C. The largest average crystallite size was owned by ZnO/Ag at annealing temperature of 300 °C which was 83.408 μm. The morphology obtained from a thin layer of ZnO/Ag 4% with annealing temperature of 250 °C and 300 °C is in the form of grains composed of ganglia structures. The ZnO/Ag layer with annealing temperature of 300 °C had the largest roughness level of 0.422 μm and the largest surface area of 197.233 μm. Meanwhile, the morphology of ZnO / Ag at annealing temperature of 350 °C and 400 °C did not form a ganglia structure so that the roughness level was low and the surface area was small. The larger the crystallite size, the higher the roughness level, and the larger the resulting surface area. ©2018 JNSMR UIN Walisongo. All rights reserved.

Keywords: ZnO/Ag, annealing, morphology, crystal structure

1. Introduction

The Environment Agency stated that pollution in the city of Semarang has entered an

alarming phase. The E. coli bacteria found in Semarang River were above the specified quality standard. The impact of water contaminated with E. coli bacteria is very dangerous if consumed by humans which attacks the

digestive organs such as dysentery and cholera. Therefore, the Government needs to take firm action against all environmental problems (Agung, 2010).

Photocatalysts are generally defined as chemical reaction processes that are assisted by light and solid catalysts (Haque, 2012). The photocatalyst process occurs when the energy from the given light matches the band gap energy of the semiconductor material, so that a chemical transformation will occur so that it converts the surrounding organic compounds into water and carbon dioxide (Fujishima, 2000). Photocatalysis is currently a promising technique for water purification compared to other conventional methods (Baruah, 2012). Photocatalyst oxidation with semiconductor catalysts can degrade organic compounds by converting them to CO₂ and H₂O (Rohmawati, 2006).

One of the semiconductors that can be used in photocatalyst applications is Zinc Oxide (ZnO). Zinc Oxide (ZnO) is an n-type semiconductor material with a band gap of 3.37 eV and a binding energy of 60 MeV (Duan, 2006). ZnO provides many advantages in various applications, especially for photocatalysts and antibacterials (Amornpitoksuk, 2012). ZnO is widely used as a photocatalyst material because besides being cheap, the catalyst is also non-toxic, low corrosion, relatively easy to synthesize, and has high photocatalytic activity (Kumar, 2011). The catalytic activity of ZnO is much better than that of other materials, because ZnO can absorb light in a wider spectrum than other materials. The catalytic activity is largely influenced by the dose of the catalyst, the concentration of the reactants, the lighting time, the intensity of the lighting, the pH value and the atmosphere (Meng, 2008). So that various studies on ZnO are now increasingly being looked at by researchers.

The crystal structure of ZnO consists of three forms, namely: the hexagonal wurtzite, cubic zinc blende, and cubic rocksalt. Of the three crystal forms, the most common is hexagonal wurtzite, and the structure of this wurtzite is more stable at room temperature (Morkoc, 2009).

ZnO is a multipurpose functional material that has various growth morphological groups, such as nanocombs, nanorings, nanohelices/nanosprings, nanobelts, nanowires and nanocages. This unique nanostructures clearly indicate that ZnO is perhaps the richest family of nanostructures among all materials, both in structure and properties. These nanostructures can be formed by various deposition methods. This new application of ZnO nanostructures is very useful in optoelectronics, sensors, transducers, photocatalysts and biomedical sciences because the materials are environmentally friendly (Wang, 2004).

Several attempts to increase photocatalytic activity are through doping with metals or non-metals or forming composites such as semiconductors/semiconductors, semiconductors/polymers or semiconductors/metals. Inserting transition metal nanostructures into ZnO is an effective method for adjusting the ZnO energy level. By increasing the doping concentration of transition metals in ZnO, their energy levels will change so that they can increase their physical and optical properties (Chauhan, 2010).

Semiconductor modifications with precious metals have attracted significant attention because they enhance the reduction process and thus also enhance the photocatalytic degradation process. Among the precious metals, Silver (Ag) was chosen because of its tremendous potential as a catalytic, non-toxicity and relatively cost-effective. This ingredient also exhibits antibacterial activity. Ag can trap the photogeneration of electrons from semiconductors and allow holes to form hydroxyl radicals that result in the current degradation of organic species (Saravanan, 2013).

Doping silver (Ag) in ZnO can increase photocatalytic activity (Zhi-Gang, 2012). In addition, the advantages of silver (Ag) as doping are its antibacterial properties, which are used to control bacterial growth in various applications (Kim, 2007). So that in this study silver (Ag) is used as a doping of ZnO to purify water by killing the bacteria in it.

One of the ZnO Ag doping thin film deposition methods is the sol-gel method. The

technique of making ZnO doping Ag with the sol-gel method has been done before with various variations of mole concentrations, obtained from the surface morphology of ZnO doping Ag, which is the formation of ZnO ganglia structures with Ag grains growing (Anggita, 2014). The optimum concentration of Ag doping was 4 mol% with the largest grain size and surface roughness (Anggita, 2016). The best photocatalytic activity of ZnO doping Ag was obtained at a concentration of 4% mole, marked by a decrease in the number of *E. coli* bacteria with a degradation percentage of 99.99951% (Anggita, 2018).

In this study, an experiment was carried out on making ZnO films with doping silver (ZnO/Ag) using a spray coating technique at annealing temperature variations of 250, 300 °C, 350 °C and 400 °C to obtain the microstructure.

2. Experiments Procedure

The research carried out was generally divided into 3 stages, namely: the process of making 4% ZnO/Ag sol gel, 4% ZnO/Ag deposition process with variations in annealing temperature, and characterization of the ZnO / Ag layer using the X-Ray Diffraction (XRD) test and Scanning Electron Microscope (SEM) test.

The process of making ZnO/Ag solution was carried out using the sol-gel method. The mechanism for making the solution, namely; Zinc Acetate in hydrate ($Zn.(COOCH_3)_2.2H_2O$) was dissolved in 2-Propanol ($CH_3CH(OH)CH_3$) at room temperature with a concentration of 0.3 M Zinc Acetate. Then MEA was dropped into a solution and stirred using a magnetic stirrer at a temperature of 70°C for 30 minutes, so that it became a ZnO solution. To get the ZnO/Ag solution, the ZnO solution was then added with Ag doping with a concentration of 4 mol% and the stirring process was continued for about 30 minutes until a homogeneous 4% ZnO/Ag solution was obtained.

The deposition of a thin layer of ZnO/Ag 4% on a glass substrate uses a spray coating technique. Before the deposition process, the glass substrate is cleaned first with the RCA (Radio Corporation of America) method, namely the glass is washed with acetone and methanol

for 10 minutes with an ultrasonic washing system to remove organic impurities such as grease and oil. Furthermore, the glass was washed with aquabides for 8 minutes and dried with a compressor.

For annealing, the temperature variations are 250 °C, 300 °C, 350 °C, and 400 °C. The dry glass substrate was placed on a hot plate with a temperature of 250°C for 10 minutes, then at the same temperature sprayed evenly with the ZnO/Ag solution. The same treatment is given for different annealing temperatures. After the deposition process, the layers were left to stand for 1 hour before the temperature finally decreased slowly to room temperature. For sintering at temperatures of 400 °C for 1 hour.

The microstructure test results of the ZnO/Ag layer with X-Ray Diffraction (XRD) resulted in crystalline peaks at two theta angles. The data obtained were used to determine the crystal structure and crystallite size. The results of the surface morphology of ZnO/Ag were obtained using a Scanning Electron Microscope (SEM). The data obtained were used to determine the surface shape and surface roughness and ZnO/Ag surface area.

3. Result and Discussion

A thin layer of ZnO doping Ag with annealing temperature variations of 250 °C, 300 °C, 350 °C, 400 °C has been successfully positioned on a glass substrate measuring 2.54 cm x 7.62 cm x 0.1 cm using a thermal spray coating technique for 1 hour and sintered at 400 °C for 1 hour as shown in Figure 1. After the ZnO: Ag layer was formed then characterized using X-Ray Diffraction (XRD) to determine the crystal structure and crystal size of ZnO/Ag and used the Scanning Electron Microscopy (SEM) test to determine the morphology of the ZnO layer with the addition of Ag doping with annealing temperature.

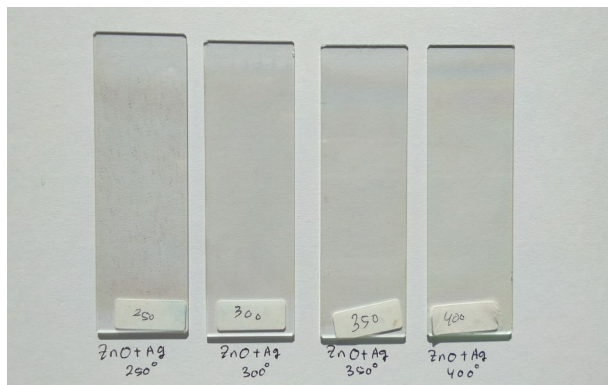


Figure 1. ZnO layer: Ag grown on a glass substrate with a variety of % Ag (a) blank, (b) ZnO, (c) 2%, (d) 3%, (e) 4% and (f) 5%

Crystal Structure of ZnO / Ag

The crystal structure of the ZnO / Ag thin film formed was observed from the XRD test results. The XRD patterns of all thin films that were deposited with variations in annealing temperature showed a peak. Figure 2 shows the diffraction pattern of the thin films deposited with various annealing temperatures.

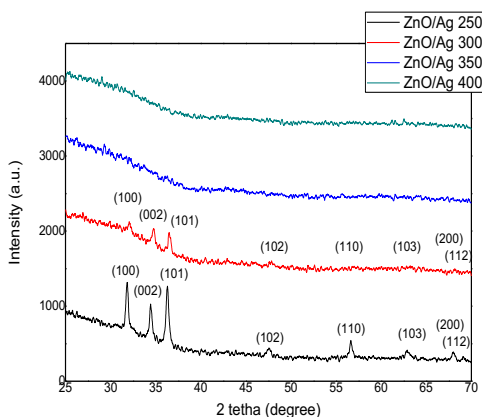


Figure 2. XRD pattern of 4% ZnO / Ag coating with variations in annealing temperature

The crystal structure of the ZnO/Ag thin film formed was identified using Origin Pro. In the 4% ZnO / Ag sample with an annealing temperature of 250 °C there are five high peaks, namely at $2\theta = 31.82^\circ$, $2\theta = 34.46^\circ$, $2\theta = 36.21^\circ$, $2\theta = 47.53^\circ$, and $2\theta = 56.62^\circ$. Each peak indicates the ZnO (100), ZnO (002), ZnO (101), ZnO (102), and ZnO (110) fields. The peaks indicate the

crystal peak pattern of the hexagonal wurtzite ZnO. Whereas in the sample ZnO / Ag 4% with annealing temperature of 300 °C there are four peaks, namely at $2\theta = 31.94^\circ$, $2\theta = 34.71^\circ$, $2\theta = 36.41^\circ$, and $2\theta = 47.82^\circ$. The respective vertices indicate the ZnO (100), ZnO (002), ZnO (101), and ZnO (102) fields. The peaks also indicate the crystal peak pattern of the hexagonal wurtzite ZnO. The appearance of these peaks indicates that 4% ZnO/Ag at annealing temperature of 250 °C and 300 °C has a hexagonal wurtzite crystal structure. The crystal structure in this research is in accordance with the research conducted by Chauhan (2010) which has obtained the ZnO/Ag crystal structure synthesized by the co-precipitation method is the hexagonal wurtzite structure. However, the ZnO/Ag samples with annealing temperature of 350 °C and 400 °C did not have crystal peaks. This shows that the ZnO / Ag microstructure at temperatures above 350 °C is amorphous. The hexagonal wurtzite ZnO structure can be described as a combination of alternating hexagonal-close-packed (hcp) sub-grids, where each sub-grid consists of one type of atom (for example Zn atoms) alternating with other types of atoms (O atoms) along the c axis. Each sub-grid includes four atoms per unit cell, each Zn atom surrounded by four O atoms and vice versa. ZnO lattice parameters for the wurtzite structure at a temperature of 300 K are $a = 3.2495 \text{ \AA}$ and $c = 5.2069 \text{ \AA}$ (Dengyuan, 2005).

It can be seen from table 1 that the ZnO/Ag thin layer with annealing temperature of 250 °C has varied crystallite sizes with different diffraction plane orientations. The size of this crystal depends on the size of the FWHM and the angle of diffraction. The higher the peak produced, the better the crystallinity is indicated by the smaller the crystal size. It can be seen from FIG. 2 that the diffraction plane 101 has the highest peak and is shown in Table 1 having the smallest crystal size. It was obtained that the average crystal size of ZnO/Ag with annealing temperature of 250 °C was 26.362 nm.

Table 1. Crystallite size of ZnO / Ag thin layer with annealing temperature of 250 °C

Diffracti on Field	2θ (°)	FWHM (°)	β (rad)	D (nm)
(100)	31,815	0,279	0,00485	29,348
(002)	34,458	0,290	0,00504	28,442
(101)	36,210	0,349	0,00607	23,758
(102)	47,536	0,349	0,00607	24,640
(110)	56,624	0,349	0,00608	25,620
D is Average				26,362

Table 2. Crystallite size of ZnO/Ag thin layer with annealing temperature of 250 °C

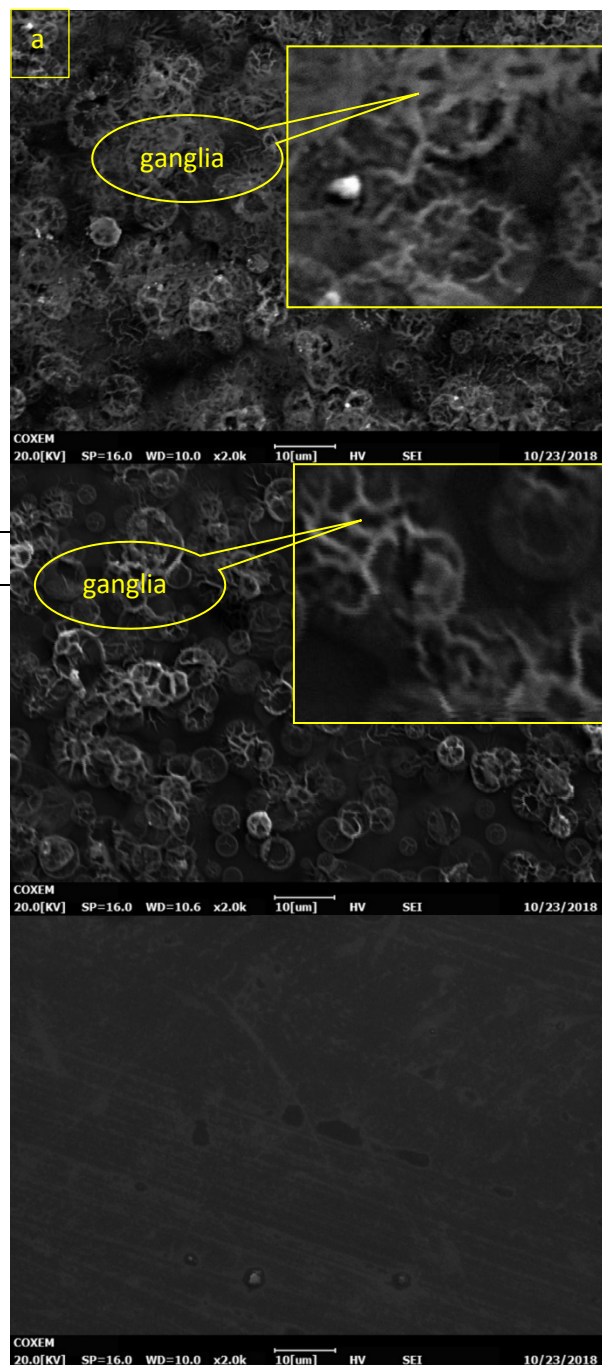
Diffracti on field	2θ (°)	FWH M (°)	β (rad)	D (nm)
(100)	31,943	0,1	0,00174	81,967
(002)	34,705	0,1	0,00174	82,568
(101)	36,414	0,1	0,00174	82,916
(102)	47,827	0,1	0,00174	86,182
D is average				83,408

Table 2 shows the ZnO/Ag thin layer with annealing temperature of 300 °C has an average crystallite size of 83.408. From table 2, it can be seen that FWHM ZnO/Ag at annealing temperature of 300 °C has decreased compared to ZnO/Ag at annealing temperature of 250 °C. This decrease in FWHM had an impact on the crystallite size. It was found that the crystallite size at annealing temperature 300 °C was greater than that at 250 °C annealing temperature. The amount of crystallite size has increased with the annealing temperature increase with the annealing temperature limit of 300 °C. However, when the annealing temperature is ≥300 °C, no crystals are formed, which is indicated by no crystal peaks. The smallest crystallite size was in the ZnO / Ag sample at an annealing temperature of 250 °C, and the largest crystallite size in the ZnO / Ag sample at an annealing temperature of 300 °C.

Surface Morphology of Coating ZnO/Ag

The 4% ZnO/Ag thin film with various variations of annealing temperature that has

been obtained from the study was then carried out with a Scanning Electron Microscope (SEM) test to determine the surface morphology of the 4% ZnO/Ag thin film with variations in annealing temperature as shown in Figure 3 with a magnification of 2,000 times.



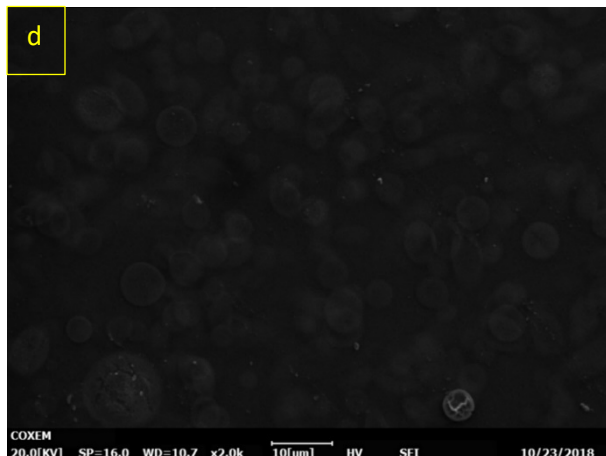


Figure 3. SEM image of 4% ZnO/Ag layer with annealing temperature (a) 250°C, (b) 300°C, (c) 350°C, (d) 400°C

Figure 3 (a) ZnO/Ag 4% with annealing temperature of 250°C and (b) annealing temperature of 300°C shows grains and each grain is composed of ganglia structure. These ganglia are indicated as ZnO ganglia. The morphological results in this study are in accordance with previous research conducted by Pusvitasari (2012) (figure 29) and Kaneva (2014) (figure 30) that the ZnO film deposited on a glass substrate has a ganglia-like structure. The results of this study showed that the morphology of the ZnO/Ag layer with annealing temperatures of 250°C and 300°C which was composed of a ganglia structure had similarities with the results of research conducted by Anggita (2014) that the morphology of the ZnO/Ag layer of 4% formed a ganglia structure with grains on the the surface indicated ganglia as ZnO and grains as Ag.

The grains formed by ZnO/Ag were due to the current method of depositing the ZnO/Ag layer with the thermal spray coating technique. The microstructure of thermal spray coating consists of many overlapping thin layers and particles with a flat base which are often referred to as splashes, so that the morphology formed is in the form of spheres. Generally, the higher the particle size, the faster the coating process, resulting in a denser and more bonded layer, both cohesively (splash-to-splash) and adhesive (layer-to-substrate) (Tucker, 2013).

The morphology of the sample surface in the form of ganglia will affect the roughness of the sample surface. The following figure 4 is a 3D image pattern of a ZnO/Ag thin layer with various variations of annealing temperature with an area of 10 x 10 m².

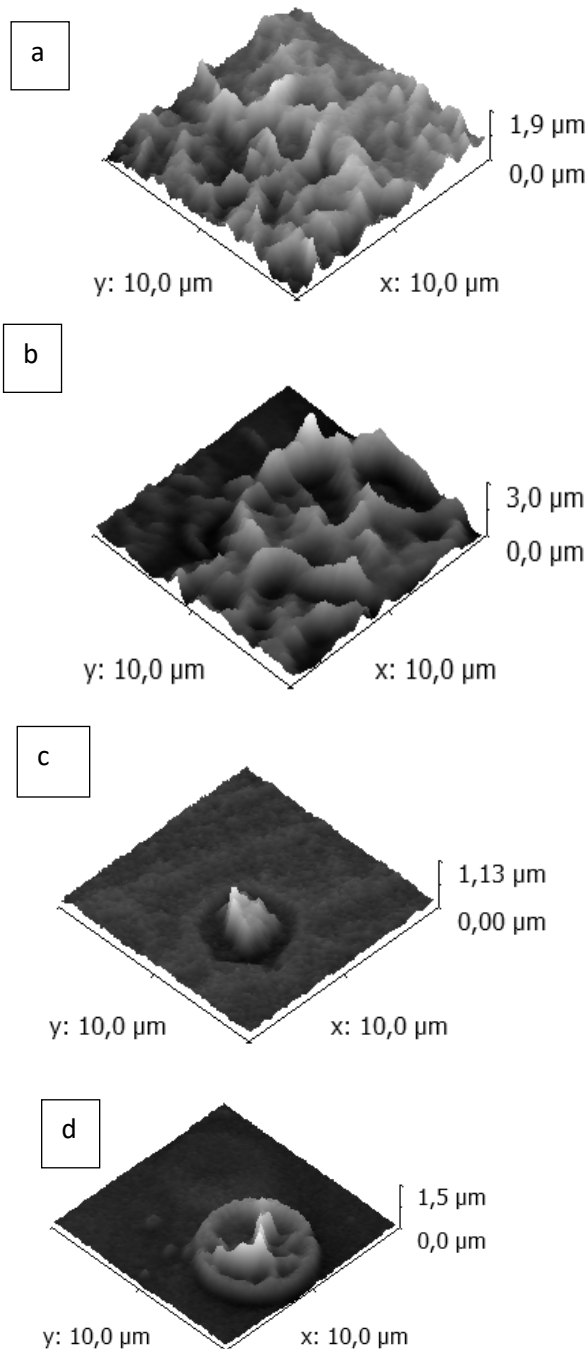


Figure 4. Three-dimensional view of the surface of a 4% ZnO/Ag layer with annealing temperature (a) 250°C, (b) 300°C, (c) 350°C, (d) 400°C

The 3D image pattern of a ZnO/Ag thin layer with various variations of annealing temperature with a scan area of 10x10µm² is shown in Figure 4. The pattern formed shows a rough morphological pattern. This is supported by the large value of the Root Mean Square (RMS) surface roughness of the 4% ZnO/Ag thin film as shown in table 3.

Table 3. RMS value and surface area of the ZnO/Ag layer with variations in annealing temperature

sample name	RMS (µm)	Surface area (µm ²)
ZnO/Ag 250°C	0,279	194,044
ZnO/Ag 300°C	0,422	197,223
ZnO/Ag 350°C	0,080	127,815
ZnO/Ag 400°C	0,158	128,121

Table 3 shows the highest level of roughness, which is 0.422 m, is found in the ZnO/Ag layer at annealing temperature of 300°C, but at 350°C the roughness value is very low. The level of surface roughness will affect the surface area. It can be seen in table 3 that the ZnO/Ag layer at annealing temperature of 300°C also has the largest surface area, on the contrary at 350°C because the roughness value is very low so that the surface area is also the lowest.

ZnO/Ag 4% with annealing temperature of 300°C has the highest RMS value when compared to other annealing temperatures, this is because the surface of ZnO/Ag 4% with annealing temperature of 300°C has a grain-shaped surface with ganglia as its constituent. The ganglia structure at 4% ZnO/Ag with annealing temperature of 300°C looks more prominent than the other samples, thus increasing the level of surface roughness. In addition, the number of cavities also increases the roughness level of ZnO/Ag at an annealing temperature of 300°C.

Compared to the morphology of the 4% ZnO/Ag layer with annealing temperature of 250°C, it also has a lot of ganglia structure, but it looks more uniform and the porosity is smaller so that the roughness value is smaller than ZnO/Ag at 300°C annealing temperature. This is like research that has been done by You (2008) that the larger the particle size will increase the level of roughness of the surface thereby

increasing the surface area. The increased surface area can be attributed to photocatalyst activity, with a large surface area resulting in more oxygen vacancies thereby increasing the diffusion between organic or inorganic molecules with the catalyst (Chang, 2013).

This surface area is also affected by the substrate treatment prior to coating by cleaning and heating, this can activate the substrate surface by increasing the free surface energy and also offers the benefit of an increased surface area for bonding the sprayed particles. Heat is transferred to the particles, so that the particles shrink, harden and bond with the rough substrate material due to the adhesion of the coating. Heating during the spray process helps the diffusion of particles into the substrate and can increase the bonds that occur between particles so that the porosity decreases (Sulzer, 2013). Therefore, the surface morphology of the ZnO/Ag annealing temperature of 250°C and 300°C has a large roughness and surface area.

ZnO/Ag has the lowest roughness level with a temperature of 350°C which is 0.080 m. The low level of roughness in ZnO/Ag with a temperature of 350°C is due to the very thin surface layer so it is estimated that the layer is not perfectly deposited on the substrate. The morphology that appears is made possible by the morphology of the glass substrate. In contrast to the case with ZnO/Ag with a temperature of 400°C, the grains are visible without any ganglia structure. The grain size decreases with increasing temperature. By decreasing the crystal size, it can reduce the level of surface roughness of the layer (Tsay, 2013).

4. Conclusion

The largest average crystallite size was owned by ZnO/Ag at annealing temperature of 300 °C which was 83.408 µm. The morphology obtained from a thin layer of ZnO/Ag 4% with annealing temperature of 250 °C and 300 °C is in the form of grains composed of ganglia structures. The ZnO/Ag layer with annealing temperature of 300 °C had the largest roughness level of 0.422 µm and the largest surface area of 197.233 µm. Meanwhile, the morphology of ZnO / Ag at annealing temperature of 350 °C and 400

°C did not form a ganglia structure so that the roughness level was low and the surface area was small. The larger the crystallite size, the higher the roughness level, and the larger the resulting surface area

Acknowledgment

The authors thank all parties involved in this research.

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