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The effect of ZnO – TiO_2 synthesis analysis using sol-gel method on calcination temperature of thin film as an alternative energy materials

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The effect of ZnO – TiO_2 synthesis analysis using sol-gel method on calcination temperature of thin film as an alternative energy materials

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Abstract. The manufacture of solar cells from ZnO – TiO_2 is synthesized at 400°C, 500°C, and 600°C. This research was carried out by synthesizing ZnO, TiO_2, and ZnO + TiO_2 with the sol-gel method and was ground using a stirrer at 80°C, 200 rpm for 30 minutes. In this study, the thin-film glass used was Select Micro Slides, which measures 76 x 26 mm of 1.0 /1.2 mm thick. The synthetic mixture used is 98% ethanol. Before testing, materials synthesis resulted from 2x2 mm coated thin glass was furnished for 1 hour with the calcination temperature of 400°C, 500°C, and 600°C. The results or the characterization performed on ZnO with a temperature of 400°C, has a peak point: 50 - 20. The graph described that there are two pairs of the same peak point, namely: 27 and 20. TiO_2 with a temperature of 500oC has a peak point of 300 - 20, and 20. And ZnO + TiO_2 with a temperature of 600°C has a peak point of 340 - 4. The temperature of crystal size varied; ZnO at a temperature of 400°C has a crystal size of 792.0585 AO or 79.205 nm, TiO_2 at a temperature of 500oC has a crystal size 492.10489 AO or 49.21 nm, and ZnO + TiO_2 at a temperature of 600°C has a crystallite size of 453 440 Ao or 45.34 nm. The result of the synthesis was different from the XRD characterization, wavelength, and crystal size. ZnO and TiO_2 can influence various characteristics such as; increasing peak point, wavelength, crystal size, and other effects that can provide heat or electrical energy.

1. Introduction

Solar radiation energy is an alternate energy source used for a very long time. One of the ways for the utilization of solar radiation energy is through the photovoltaic conversion system using an optoelectronic device called solar cells [1]. The first research on the photovoltaic effect was carried out by Becquerel in 1839. Becquerel detected and analyzed the presence of an electric voltage when sunlight hit the electrodes in an electrolyte solution [2]. Then in 1954, researchers from Bell Laboratories made a development of the photovoltaic effect into solar cells using silicon materials, semiconductor materials and diffused crystals, for the conversion efficiency of solar cells obtained about 4.5%, [3].

Solar cells device uses a semiconductor material as the main component. The semiconductor that is widely used in solar cells today is silicon. In solar cells, there are two processes that are mutually sustainable, namely the process of light absorption and separation of electric charges [4].

The Material metal oxide having photocatalytic properties are TiO_2 and ZnO. Both photocatalyst activity has been high in the wavelength range of UV. TiO_2 and ZnO are both photocatalysts and can degrade organic compounds. Several studies have reported that the efficiency of ZnO activity is relatively higher than TiO_2 as photodegradation in aqueous solution [5]. According to Kasuma, [6], ZnO has several advantages such as stable to light, non-toxic, environmentally friendly and the ability to decompose organic compounds and bacteria that often contaminate the environment.

Zinc oxide (ZnO) is one of the most promising semi-conductor materials to be applied to various microelectronic and opelectronic devices, including transparent thin film transistors, photo detectors and solar cells. ZnO material has direct wide band gap (3.37 eV), thus allowing direct absorption of UV radiation (band-to-band transition) [5]. There are various methods in the synthesis of ZnO such as magnetron sputtering, spray pyrolysis, electrodeposition, sol-gel, pulsed laser deposition, chemical bath deposition and others [7].

One of the simple and quite easy synthesis methods is the method of sol-gel. In this method, the synthesis of ZnO is carried out by changing several phases, namely the solution into a sol (a colloid that has suspended solids in its solution) and then into a gel phase. In this phase, the gel size is larger than that of the sol [8].

There are some aims of this research, which are, to determine the effect of the synthesis and calcination temperature on ZnO and TiO_2, to determine the effect of differences in peak point and crystal size on XRD testing on the synthesized ZnO and TiO as semiconductor materials, to determine changes in the development of sol-gel and its properties using ZnO and TiO_2 toward the glass material used, and to determine the morphological and structural changes in the SEM - EDX test which are significant and can improve the quality of ZnO and TiO_2 as alternative energy materials.

2. Literature Review

Compounds Titanium dioxideare the (TiO2) most widely applied for semiconductors in DSSC because of its large band gap , is harmless, and is inexpensive [9]. However, in its application as a semiconductor in DSSC, TiO_2 has a weakness. The characteristics of TiO_2 as an indirect band gap semiconductor in which the position of the valence band is slightly far from the conduction band so that the excitation of electrons during light absorption is less efficient [10]. Therefore, an alternative to TiO_2 is needed as a semiconductor in DSSC, such as ZnO compounds.

The use of zinc oxide (ZnO) as a semiconductor is an alternative to TiO_2, because ZnO has shown multifunctional properties with high binding energy (60 MeV), low resistivity, non-toxicity, high transparency in the visible range-absorbing, and large light absorption [11].

In addition, ZnO also exhibits band gap a wideof 3.3 eV with high optical transparency at room temperature, the ability to bind 60 MeV free electrons, high resistivity (10-4 – 1012 .cm), and electron mobility. by 200 cm2.v-1.s-1 [12]; [13]; [14]. Another advantage of ZnO is that it has a very high chemical stability, high electrochemical coupling coefficient, has a broad UV absorption capability, and is also very sensitive to light [15].

ZnO semiconductor is known to be stable at room temperature and withstand very high temperatures [16]. Therefore, in this study, the synthesis of ZnO and TiO was carried out as semiconductors, and the method sol-gel was used in this study.

3. Research Methodology

This research was conducted at the Department of Physics and Chemistry, State University of Medan, In this research, there were three steps that will be carried out to synthesize ZnO, TiO_2, ZnO + TiO_2 . The first stage is ZnO was weighed 0.5 grams, then mixed with 2 ml of 98% ethanol, then the sample is ground using a stirrer and added mortar as a stirrer. The duration of the synthesized ZnO semiconductor material was 1 hour at a speed of 500 rpm at 80^{o}C calcination. Likewise ,the synthesis treatment was the same for TiO_2.

The second stage is to synthesize a mixture of ZnO + TiO_2, weighing 0.5 grams and added with 3 ml of 98% ethanol. The treatment for synthesizing this semiconductor material is the same as for ZnO and TiO_2 which was ground with a Stirrer apparatus at a temperature of 80^{o}C and a speed of 500 rpm for 1 hour.

After completion of the synthesis which resulted in the treatment sol gel, then each material was sol gel attached to thin film or glass preparations measuring 2 cm x 2 cm, then spin coating with a time of 30 seconds and a speed of 1000 rpm. Then each finished sample in the spin coating was then purified for 1 time using a Nabertherm made in Germany with each calcination of 400°C, 500°C and 600°C. After completion of calcination then the samples allowed to stand for 30 minutes at room temperature. The third step is to characterize each sample with XRD and SEM-EDX as shown in the following semiconductor materials:
1. ZnO with a calcination temperature of 400°C
2. TiO_2 with a calcination temperature of 500°C and
3. ZnO + TiO_2 with a calcination temperature of 600°C

4. Result and Discussion

4.1. The calculation results to determine the crystal size of the highest peak with variations in temperature and holding time can be calculated as follows
Calculating the size of the crystal with Debye Scherrerequation:

$$D = \frac{0.9\lambda}{\beta cos\theta} \quad (1)$$

By:
D = the crystal size (nm)
λ = wavelength(Å)
β = *FWHM* (Full*Width Half*Maximum)
θ = diffraction angle

a. ZnO 400°C
Sick:
2= 44.4973
= 0.14130; 1°= 0.01745
= 1.541874Å
then:

$$D = \frac{0.9\lambda}{cos\theta}$$
$$= \frac{0.9(1.541874)}{(0.14130)(0.01745)\ cos\ cos\ \left(\frac{44.4973}{2}\right)}$$
$$= \frac{1.3876866}{(0.002465)(0.71328)}$$
$$= \frac{1.3876866}{0.0017582}$$
$$= 792.0585Å$$
$$= 79.205nm$$

4.2. TiO2 500°C
Known:
2= 44.4951
= 0.17460°; 1°= 0.01745
= 1.541874Å

$then$:

$$D = \frac{0.9\lambda}{cos\theta}$$
$$= \frac{0.9(1.541874)}{(0.17460)(0.01745)\ cos\ cos\ \left(\frac{44.4951}{2}\right)}$$
$$= \frac{1.3876866}{(0.00304677)(0.92555669)}$$
$$= \frac{1.3876866}{0.0028199}$$
$$= 492.10489 Å$$
$$= 49.21 nm$$

4.3. ZnO-TiO₂ 600°C

Known:
2= 44.5115
= 0.18950°; 1°= 0.01745
= 1.541874Å
$then$:

$$D = \frac{0.9\lambda}{cos\theta}$$
$$= \frac{0.9(1.541874)}{(0.18950)(0.01745)\ cos\ cos\ \left(\frac{44.5115}{2}\right)}$$
$$= \frac{1.3876866}{(0.0033067)(0.92550249}$$
$$= \frac{1.3876866}{0.00306035}$$
$$= 453.440 Å$$
$$= 45.34 nm$$

From the calculation results, it can be seen that the crystal size and the highest peak with variations in calcination temperature and holding time can be seen in the following table:

Table 1. Calcination temperature.

No	Material	Calcination Temperature	(Amstrong)	Crystal Size (nm))
1	ZnO	400^{O}C	792.0585	79.205
2	TiO_2	500^{O}C	492.1049	49.21
3	ZnO + TiO_2	600^{O}C	453.4400	45.34

From the calculation table above, the ZnO material, with its 400°C calcination temperature had the highest result number 1 which is ƛ = 792.0885 Ao and 79.205 nm crystal size. The second highest is TiO_2 by calcination temperature of 500°C where ƛ = 492 and 49.21 crystal size, and the third place which have the highest value is ZnO + TiO_2 with a calcination temperature of 600°C, i.e. = 453,4400 Ao and a crystal size of 45.34.

4.4. The results of characteristic testing on XRD

4.4.1. XRD testing of ZnO with a calcination temperature of 400°C. In the graph of ZnO above, the calcination temperature of 400°C has a peak point ak : 50, 43, 30, 27, 27, 23, 20 and 20. In this graph, there are two pairs of the same peak, namely: 27 and 20.

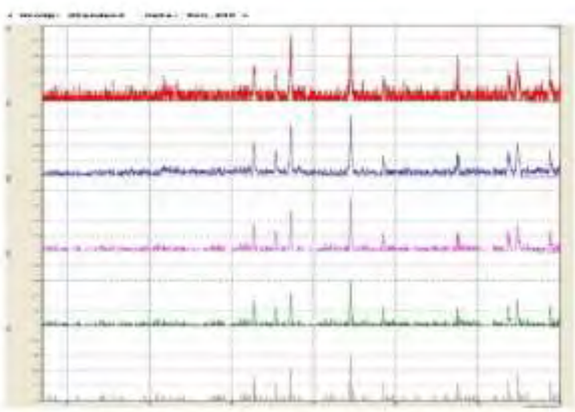

Figure 1. Calcination temperature of 400oC

4.4.2. XRD testing of TiO_2 with a calcination temperature of 500°C. In the graph above, TiO_2 calcined at a temperature of 500°C, has peak points of 300, 160, 60, 40, 20 and 20.

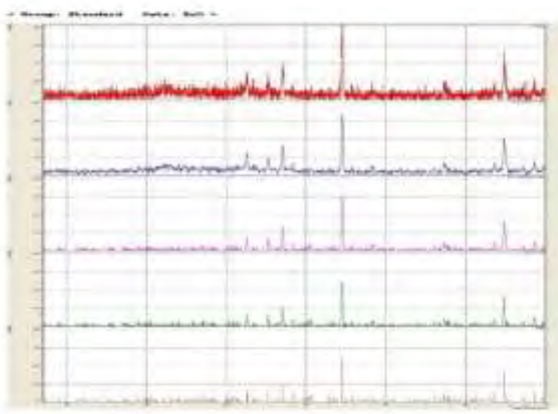

Figure 2. Calcination temperature of 500°C

4.4.3. XRD testing of ZnO + TiO_2 with a calcination temperature of 600°C. In the graph bellow ZnO + TiO2 which is calcined at a temperature of 600°C has peak point of 340, 190, 6 and 4.

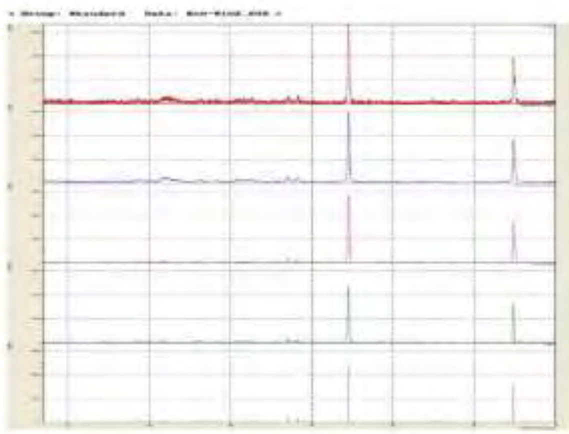

Figure 3. Calcination temperature of 600°C

From the three characteristic tests of XRD, it was found that ZnO + TiO_2 with a calcination temperature of 600°C has the highest peak point, which is 340. Followed by TiO_2 with a calcination temperature of 500°C, which is 300 and ZnO with a calcination temperature of 400°C, which is 50. They are described in the following table:

Table 2. Describing table.

No	Semiconductor Material	Temperature (°C)	Peak Point
1	ZnO + TiO_2	600	340
2	TiO_2	500	300
3	ZnO	400	50

5. Conclusion

From the results of the study, it was found that the ZnO semiconductor composite at a temperature of 400°C had a wavelength of 792.0585 AO and a crystal size of 79.205 nm. At a temperature of 500 °C wave length 492.1049 AO and the crystal size of 49.21 nm. And at 600 wave length 453.4400 AO and the crystal size of 45.34 nm. . In the X-ray test, the highest and cancel peaks are found at a temperature of 600°C.

Based on the data and the results of the research, that the Influence of the synthesis process ZnO-TiO2 by calcination temperature of the sol-gel method, can increase the advantage in modifying the pattern nanoparticles through simple process conditions. And gives the optimum value at a gel temperature of 400°C.

In the process of making ZnO, TiO and ZnO + TiO_2 composite semiconductor materials or a mixture of both, we must first arrange how many grams of each mixture we use, the duration of heating as well as the tolerance and overall incorporation, in order to avoid errors in the formation of the gel sol, as well as the calcination process. Also, to complete the process of forming the morphology of the ZnO-TiO_2 semiconductor, it is necessary to adjust the pH in the sol-gel process.

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