

## Research Article

# Influence of the Hydrothermal Method Growth Parameters on the Zinc Oxide Nanowires Deposited on Several Substrates

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Received 6 August 2014; Revised 21 October 2014; Accepted 4 November 2014; Published 18 November 2014

Academic Editor: Shadi A. Dayeh

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We report the synthesis of ZnO nanowires grown on several substrates (PET, glass, and Si) using a two-step process: (a) preparation of the seed layer on the substrate by spin coating, from solutions of zinc acetate dihydrate and 1-propanol, and (b) growth of the ZnO nanostructures by dipping the substrate in an equimolar solution of zinc nitrate hexahydrate and hexamethylenetetramine. Subsequently, films were thermally treated with a commercial microwave oven (350 and 700 W) for 5, 20, and 35 min. The ZnO nanowires obtained were characterized structurally, morphologically, and optically using XRD, SEM, and UV-VIS transmission, respectively. XRD patterns spectra revealed the presence of Zn(OH)<sub>2</sub> on the films grown on glass and Si substrates. A preferential orientation along *c-axis* directions for films grown on PET substrate was observed. An analysis by SEM revealed that the growth of the ZnO nanowires on PET and glass is better than the growth on Si when the same growth parameters are used. On glass substrates, ZnO nanowires less than 50 nm in diameter and between 200 nm and 1200 nm in length were obtained. The ZnO nanowires band gap energy for the films grown on PET and glass was obtained from optical transmission spectra.

## 1. Introduction

Zinc oxide (ZnO) is an interesting material due to its excellent optical, chemical, electrical, and piezoelectric properties [1–4]. ZnO is an n-type oxide semiconductor material and has been investigated since it has very stable thermal characteristics, with a direct wide band gap of 3.37 eV, a large exciton binding energy of 60 meV, and transparent conductivity. ZnO nanowires are one dimensional (1D) structures with remarkable physical properties. Comparing with carbon nanotubes, ZnO nanowires have a good mechanical strength and thermal and chemical stability; thus they are potential substitute of carbon nanotubes. ZnO nanowires are widely applied in the field of optoelectronic and nanomechanics, such as in ultraviolet light-emitting diodes, varistors, surface acoustic wave (SAW) components, transparent electrodes for solar cell window, gas sensors, and photocatalysts [5, 6].

Physical, chemical, and electrochemical methods have been carried out for achieving ZnO nanowires; some of these are metal-organic vapor-phase epitaxy, chemical vapor deposition, and hydrothermal deposition [7, 8]. Hydrothermal method has attracted much attention in the field of ZnO nanostructures growth because of the simplicity of the required equipment in the growth process and the possibility of low temperature implementation [9]. In that work, ZnO well aligned single crystalline hexagonal nanowires of 100–200 nm wide at 95°C were obtained. A disadvantage of this method is the time required for the synthesis of nanowires spanning from several hours to days [10]. Several methods have been suggested in order to increase the growth rate of hydrothermally grown ZnO nanowires. A microwave assisted hydrothermal method with the aim of rapid heating of the solution and so reducing the time needed for reaching the crystallization temperature in the growth environment has been carried out successfully [11].

TABLE 1: Growth parameters, XRD results, nanowire diameter, and band gap energy for samples of ZnO nanowires grown on PET, glass, and Si.

Sample	Power (W)	Heating time (min)	Highest XRD reflections	Nanowire diameter (nm)	Energy (eV)
PZO1	700	5	(002)	180–200	3.28
PZO2	700	20	(002)	90	3.28
PZO3	700	35	(100)	80–100	3.28
PZO4	350	5	(100), (002), (101)	140	3.29
PZO5	350	20	(100)	90	—
PZO6	350	35	(100)	180	3.28
GZO1	700	5	(002)	200	3.27
GZO2	700	20	(100), (002)	200	3.22
GZO3	700	35	(100), (002), (101)	100–200	3.33
GZO4	350	5	(100)	<25	3.33
GZO5	350	20	(100), (101)	200–600	—
GZO6	350	35	Not preferred orientation	50–100	3.22
SZO1	700	5	(002)	80–200	—
SZO2	700	20	(100), (002), (101)	70–200	—
SZO3	700	35	(101)	70–200	—
SZO4	350	5	(100)	200–400	—
SZO5	350	20	—	—	—
SZO6	350	35	(100)	200–400	—

In this work, ZnO nanowires are grown on several substrates (PET, glass, and Si) by hydrothermal method using the same growth parameters in order to determine the influence of the substrate in the vertical alignment of the ZnO nanowires. To obtain ZnO nanowires efficiently a commercial microwave oven for rapid heating of the material was used. With this procedure the processing time for growing ZnO nanowires can be reduced up to 1 min [12].

The ZnO nanowires were characterized structurally, morphologically, and optically by X-ray diffraction, scanning electron microscopy (SEM), and UV-VIS transmission measurements at room temperature.

## 2. Materials and Methods

ZnO nanowires were grown hydrothermally on several substrates such as polyethylene terephthalate (PET), glass, and p-type Si (100) based on the method developed by Unalan et al. [11]. This method consists in two-step process: (a) preparation of the ZnO seed layer and (b) growth of the nanostructures. First step: solutions of zinc acetate dihydrate and 1-propanol of 10 mM concentration were spin coated on the substrate at 2000 rpm for 54 s at room temperature using a syringe pump; then the substrate was annealed at 100°C for 60 s.

A ZnO seed layer was obtained after three of such cycles. Second step: vertical ZnO nanowires were grown by dipping the substrate in a 25 mM equimolar solution of zinc nitrate hexahydrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) and hexamethylenetetramine (HMTA) solution in deionized water. Subsequently, films were thermally treated with a commercial microwave oven at different power settings (350 and 700 W) for 5, 20, and 35 min. Finally, the substrates were taken out from the growth solution, rinsed with deionized water, and

dried under nitrogen flow. Prior to spin coating, the substrates were cleaned by sonication in acetone and isopropanol for 12 min and dried in a nitrogen flow.

The morphological, structural, and optical properties of the ZnO nanowires were studied by scanning electron microscopy (SEM), X-ray diffraction (XRD), and optical transmission (OT), respectively. SEM system was operated at 5 kV in order to obtain the ZnO nanowire images with a secondary electron detector in a Zeiss MOD Auriga 39-16 microscopy. The X-ray diffraction patterns were measured in Grazing-incidence small-angle X-ray scattering (GISAXS) with Xpert PRO Diffractometer from PANalytical using Cu radiation ( $K_\alpha = 1.54 \text{ \AA}$ ). Optical transmission measurements were obtained with a Spectrometer UV/VIS Lambda 35 Perkin-Elmer in the region of 300 nm to 1100 nm. Table 1 shows the main growth parameters (power of microwave oven and heating time) used during the growth of ZnO nanowires on different substrates. The first letter of the sample names referred to the substrate used; P for PET, G for glass, and S for Si.

## 3. Results and Discussion

**3.1. XRD Patterns.** Figures 1, 2, and 3 show Bragg-Brentano  $\theta$ -2 $\theta$ XRD scans for samples PZO1 to PZO6, GZO1 to GZO6, and SZO1 to SZO6, grown at different conditions of thermal treatment on PET substrates, glass substrates, and Si substrates, respectively. The thermal treatment was performed at 350 W and 700 W for different times: 5 min, 20 min, and 35 min.

In the XRD scans of Figure 1 we identify the crystalline phase of zinc oxide (Zincite); the reflections with highest intensity are summarized in Table 1. When the furnace power

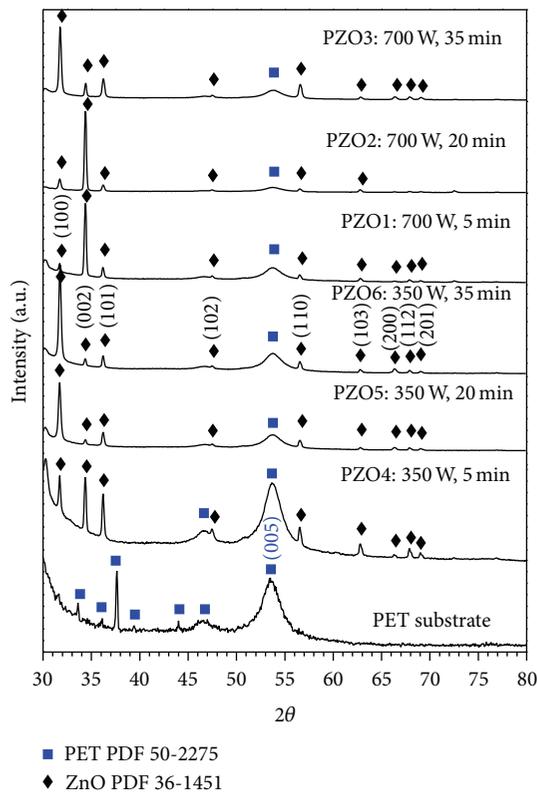


FIGURE 1: XRD profiles for  $2\theta$  in the range from  $30^\circ$  to  $80^\circ$  for all samples of ZnO nanowires on PET substrate grown at several conditions.

was 350 W and the treatment time was 20 min and 35 min, the intensity of the reflection (100) of ZnO has the highest intensity, with a preferential growing in the *a*-axis direction. When applying a heating time of 5 min, the intensity of the reflections of ZnO, in the directions (100), (002), and (101), was the highest and there was no preferential orientation. When the furnace power was increased to 700 W and the heating time was 5 min and 20 min, the highest intensity was obtained for the reflection (002) indicating the preferential growing in the *c*-axis direction in accordance with the results of Unalan et al. [11] and Vayssieres [9]. On the other hand, applying 35 min of heating time leads to the highest intensity in the reflection (100) with a preferential growing in the *a*-axis direction. Furthermore, the thickest film was obtained when the furnace power was 700 W and 20 min of heating time. The signal of the PET substrate can be observed even to high power and large heating time in the furnace. However, the thickness of the film is not thick enough to block the substrate signal. In the thermal treatment conditions studied, the growth of ZnO nanowires is performed preferentially in the directions (100) and (002).

The crystalline phases obtained for the growth on glass substrates (Figure 2) are ZnO and  $\text{Zn}(\text{OH})_2$ . Most of the crystalline phase reflections of  $\text{Zn}(\text{OH})_2$  are overlapped with the peaks associated to the ZnO nanowires. When the furnace power was 350 W and the treatment time was 5 min and 20 min, the intensity of the reflections of ZnO in the direction (100) has the highest intensity, with a preferential growing in

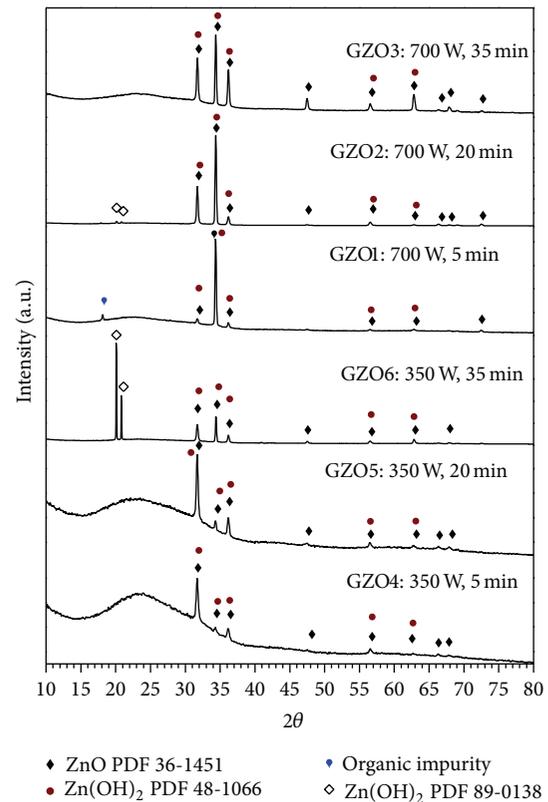


FIGURE 2: XRD profiles for  $2\theta$  in the range from  $10^\circ$  to  $80^\circ$  for all samples of ZnO nanowires on glass substrate grown at several conditions.

the *a*-axis direction. When the furnace power was increased to 700 W and the heating time was 5 min and 20 min, as in results obtained with PET, the highest intensity was obtained for the reflection (002) indicating the preferential orientation in the *c*-axis direction. When applying a heating time of 35 min, in both furnace powers, 350 W and 700 W, the intensity of the reflections of ZnO, in the directions (100), (002), and (101), was the highest and there was no preferential orientation. We can observe that the intensity of the peaks increases for a larger heating time, in particular the peaks at  $31^\circ$  (100) and  $36^\circ$  (101) for high power setting (700 W) but not at low power (350 W). There is a trace phase of an organic impurity in sample GZO1 and a  $\text{Zn}(\text{OH})_2$  phase (PDF 89-0138) around  $20^\circ$  in samples GZO2 and GZO6. It can be observed that the  $\text{Zn}(\text{OH})_2$  phase (PDF 89-0138) disappears when the power increases to 700 W and the intensity of the peaks corresponding to the ZnO nanowires increases.

As for the deposition on Si substrates as observed in Figure 3, when the furnace power was 350 W and the heating time was 35 min, we obtained a better coverage of the Si substrate since the intensity of its highest reflection decreases. We observed also this decrease when the furnace power was 700 W and for a heating time of 5, 20, and 35 min. The ZnO reflection (100) increases at low power and small time of treatment in the furnace (350 W, 5 min). For a high power (700 W), the reflections increase as a function of the heating time. This can be observed comparing the intensities of

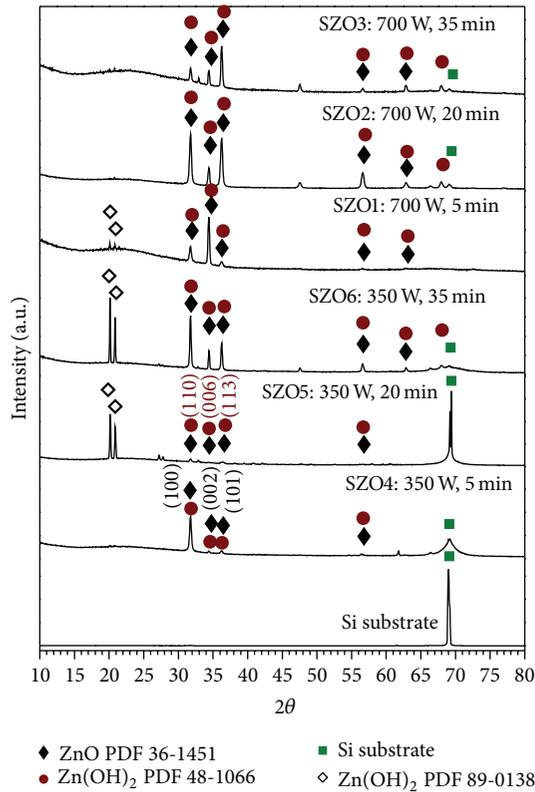


FIGURE 3: XRD profiles for  $2\theta$  in the range from  $10^\circ$  to  $80^\circ$  for all samples of ZnO nanowires on Si substrate grown at several conditions.

the  $\text{Zn(OH)}_2$  phase (PDF 89-0138). The reflection (002) increases for a heating time of 5 min indicating the preferential growing in the  $c$ -axis direction. The reflections (100) and (101) increase for a heating time of 20 min. The reflections (101) increase for a heating time of 35 min. For the case of  $\text{Zn(OH)}_2$  phase (PDF 89-0138), which appears at lower power (350 W) and for a heating time of 20 min, its relative intensity increases when the heating time increases to 35 min and decreases when the power furnace increases at 700 W and the heating time is 5 min. This reflection decreases to a trace level for higher heating times.

**3.2. Scanning Electron Microscopy.** SEM images of the ZnO nanowires arrays grown at 700 W and 350 W for (a) 5, (b) 20, and (c) 35 min on PET, glass, and Si substrates are shown in Figures 4 to 9, respectively.

The growth of the nanowires aligned vertically on the PET substrates, for heating time of 5 min and 20 min (700 W of furnace power), is clearly observed in Figures 4(a) and 4(b), besides the hexagonal cross section of the wires (wires diameters are summarized in Table 1). These are in agreement with the XRD results where the preferential growing of the (002) planes is observed. For a heating time of 5 min, the diameters vary between 180 and 200 nm approximately; for 20 min, the average diameter is 90 nm approximately. While in Figure 4(c), heating time of 35 min, the nanowires grew in random directions and the diameter, between 80 and

100 nm approximately, decreases in needle tip form; on the other hand, by XRD it is observed that the planes (100) grew preferentially. In these conditions of heating time and furnace power, increasing the time, the diameter of the nanowires decreases but it is observed their length increases. As for example, at 5 and 20 min the length is 200 nm approximately, but at 35 min the length increases to 300–400 nm. As we can see, over PET, the nanowires increased to twice the length, when the furnace power is set at 700 W. This is in agreement with our previous work [12].

We observed growth of the nanowires (Figure 5(a)) for a heating time of 5 min (350 W of furnace power) and that according to XRD results, the reflections of the planes (100), (002), and (101) were of the highest intensity. In these conditions a preferential growth was not observed and the nanowires diameter was 140 nm approximately. In Figure 5(b) the nanowires grew parallel to the substrate but randomly orientated for a heating time of 20 min (350 W). By XRD a preferential growth of the planes (100) is observed. The nanowires diameter was of 90 nm approximately. Besides, the growth at 20 min shows also ZnO structures like bars with diameters larger than the nanowires that possibly are concerned to precipitates from the source solution for the nanowires growing. In Figure 5(c) the film shows the growth of nanowires but also particles of seed-like ZnO, for a heating time of 35 min (350 W). XRD results indicate that the (100) planes grew preferentially. The nanowires diameter was of 180 nm approximately. We observed that at 350 W and heating time of 5 and 35 min, the length is of 330 nm in average, but with 20 min of heating time the diameter is 90 nm and the length of the nanowires is 660–1000 nm. However, ZnO structures like bars of 400 nm in diameter and 3500 nm in length, approximately, were obtained.

The ZnO nanowires grow vertically aligned on glass substrates at 700 W for a heating time of 5 min (Figure 6(a)). The hexagonal cross section of the wires can be clearly observed in the SEM images. This is in agreement with the XRD results where the preferential growth of the (002) planes is observed. The diameters of the ZnO nanowires are around 200 nm. For a longer heating time of 20 min the XRD reflections of the planes (100) and (002) were of the highest intensity and the nanowires diameter was 200 nm (Figure 6(b)). On the other hand, it can be observed that, for a heating time of 35 min (Figure 6(c)), the ZnO nanowires do not grow vertically but parallel to the substrate and with a length from 200 nm to 1200 nm and diameters between 100 and 200 nm. This is in agreement with XRD results where in these conditions a preferential growth of the nanowires was not observed. We observed that when the heating increases to 35 min at 700 W, the nanowire length increases significantly.

On the other hand, ZnO nanowires grown at 350 W on glass substrates for a heating time of 5 min showed a vertical alignment according to XRD results, and the reflection of the plane (100) was of the highest intensity. The ZnO nanowires were grown with diameter smaller than 25 nm (Figure 7(a)). For the sample processed at 350 W for 20 min, it can be observed that the ZnO nanowires were grown with diameters between 200 and 600 nm with no preferential growth. From the XRD results the reflections of planes (100) and (101)

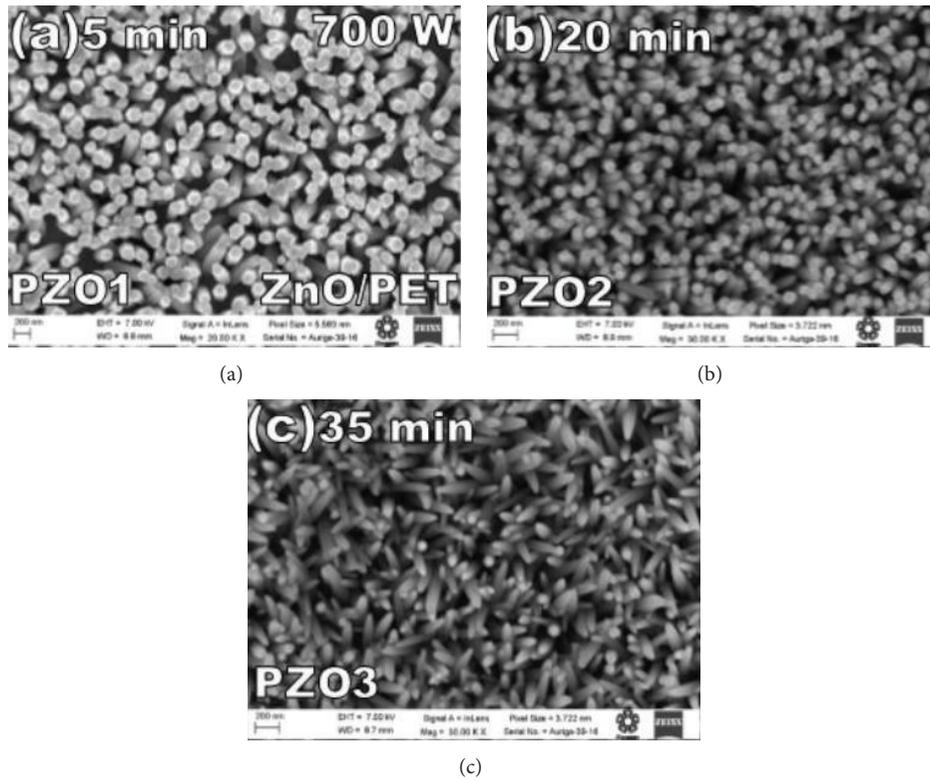


FIGURE 4: Scanning electron microscopy images of the ZnO nanowires grown on PET substrate at 700 W and with a heating time of (a) 5 min, (b) 20 min, and (c) 35 min. Scale bar of 200 nm.

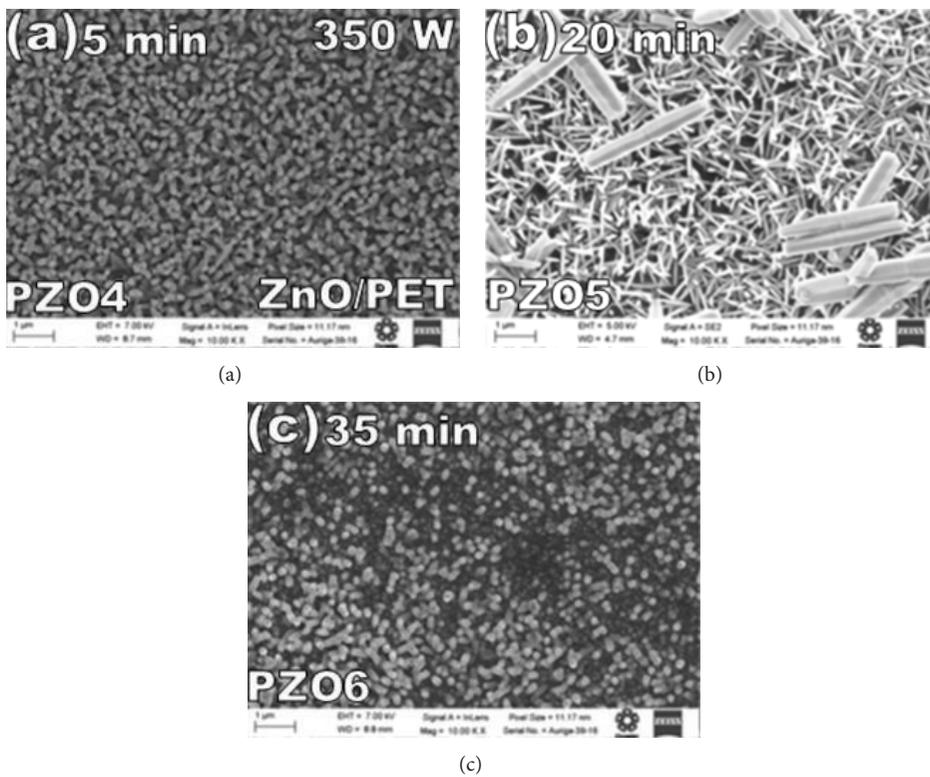


FIGURE 5: Scanning electron microscopy images of the ZnO nanowires grown on PET substrate at 350 W and with a heating time of (a) 5 min, (b) 20 min, and (c) 35 min. Scale bar of 1 μm.

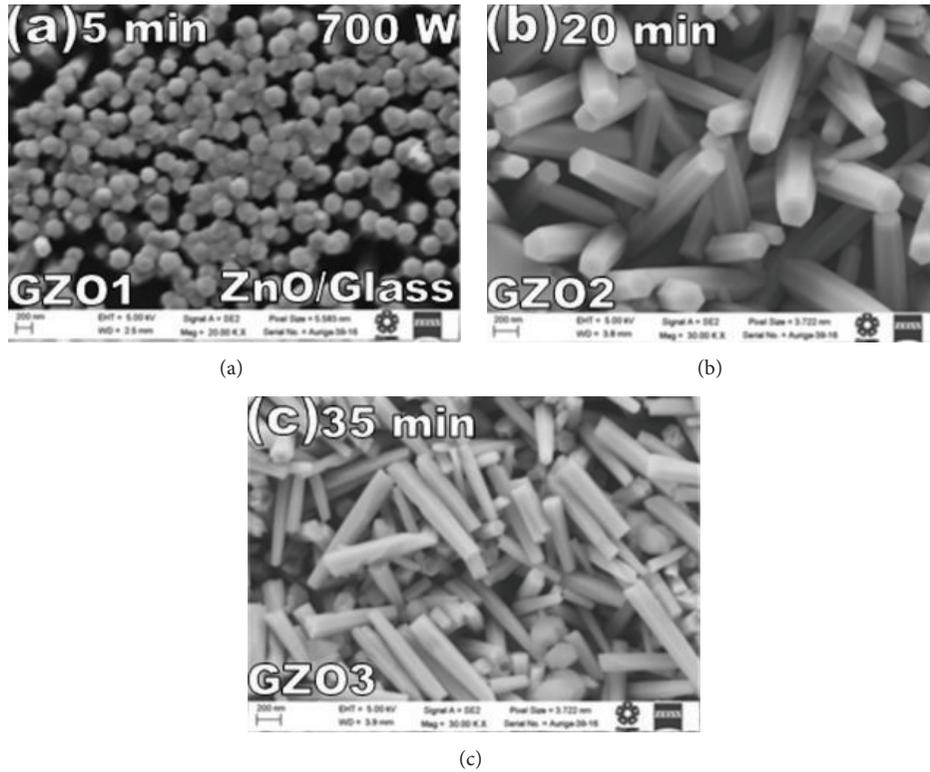


FIGURE 6: Scanning electron microscopy images of the ZnO nanowires grown on glass substrate at 700 W and with a heating time of (a) 5 min, (b) 20 min, and (c) 35 min. Scale bar of 200 nm.

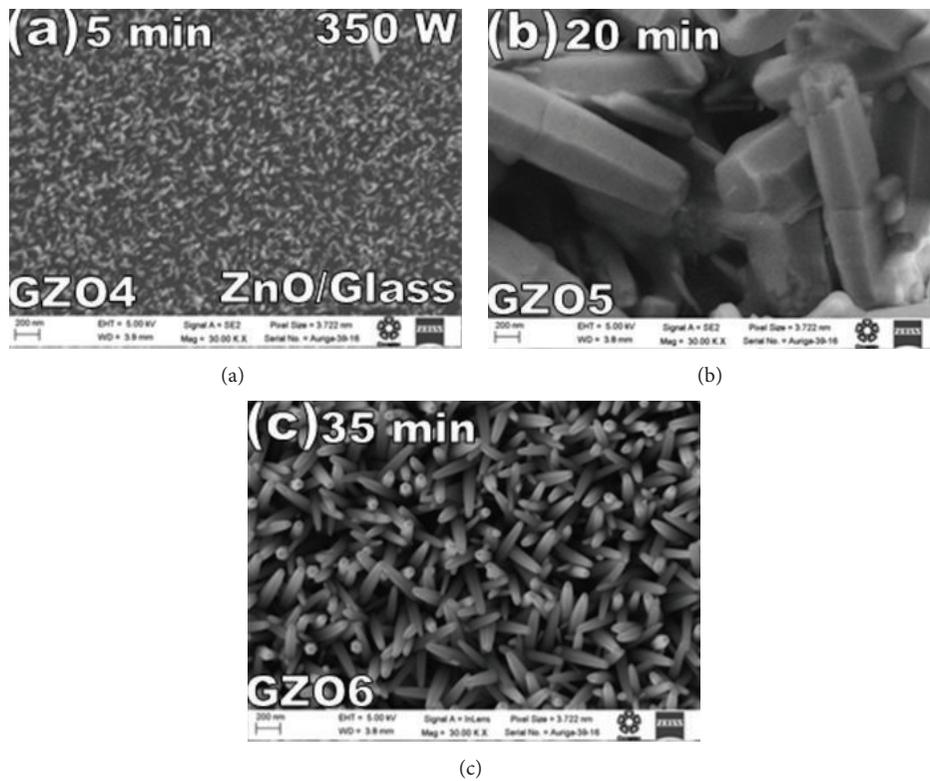


FIGURE 7: Scanning electron microscopy images of the ZnO nanowires grown on glass substrate at 350 W and with a heating time of (a) 5 min, (b) 20 min, and (c) 35 min. Scale bar of 200 nm.

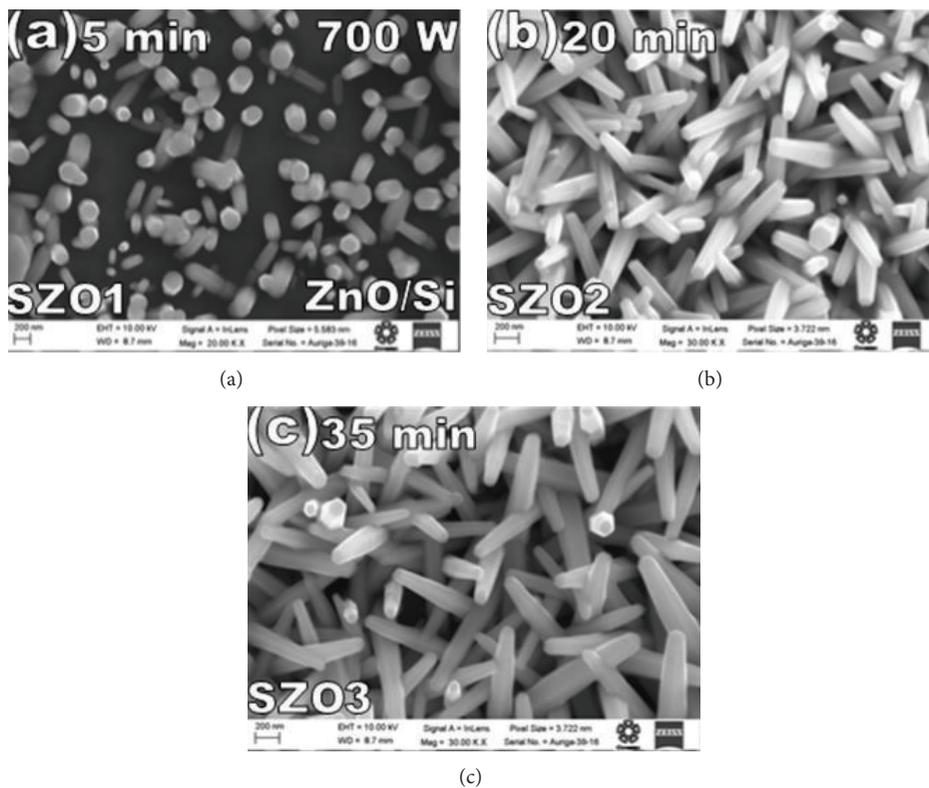


FIGURE 8: Scanning electron microscopy images of the ZnO nanowires grown on Si substrate at 700 W and with a heating time of (a) 5 min, (b) 20 min, and (c) 35 min. Scale bar of 200 nm.

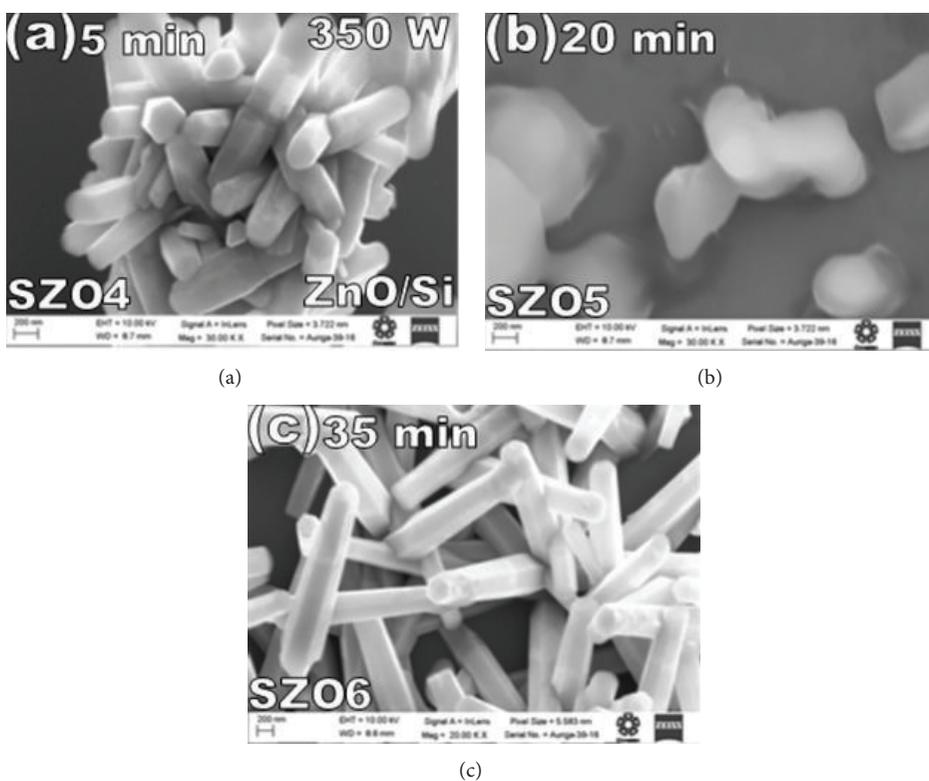


FIGURE 9: Scanning electron microscopy images of the ZnO nanowires grown on Si substrate at 350 W and with a heating time of (a) 5 min, (b) 20 min, and (c) 35 min. Scale bar of 200 nm.

were of the highest intensity (Figure 7(b)). In both samples, the XRD signal of the glass substrate was strong obtaining thin films. However, at 350 W for a heating time of 35 min (Figure 7(c)), the nanowires grew in random directions and the diameter, between 50 and 100 nm approximately, decreases in needle tip form. This is in agreement with XRD results where a preferential growth was not observed. We observed that with heating time of 5 and 35 min, the length is 100 and 400 nm, respectively. But with 20 min of heating, the length increased to 2000 nm approximately.

For the deposition on Si substrates (Figure 8), ZnO nanowires grew vertically aligned for a heating time of 5 min at 700 W, but with a low density, and according to XRD results, the reflection of the plane (002) was the highest in intensity. The diameter of these nanowires was between 80–200 nm and 450 nm of length. By increasing heating time to 20 min the nanowires grew randomly orientated, as observed in Figure 8(b). This is in agreement with the XRD results where the reflections of planes (100), (002), and (101) were of the highest intensity. The diameter of the nanowires was between 70 and 200 nm and decreases to the tip, while the length for this heating time increased to 500–900 nm. The same diameters were obtained for sample processed during 35 min, but the nanowires length increased to 600–1200 nm; see Figure 8(c). In this sample, the reflection (101) has the highest intensity.

By decreasing the furnace power to 350 W (Figure 9) we observed that, for short heating time (5 min), the ZnO nanowires are not aligned and grow as clusters where by XRD the reflection of highest intensity was the (100). The nanowires diameters were between 200 nm and 400 nm. But for 35 min (Figure 9(c)) the nanowires grow randomly although by XRD the highest intensity is for reflection (100). Their diameters were the same as those obtained for 5 min of heating time.

At 350 W and 700 W, we observed that the length of the nanowires increased with increasing heating time.

From SEM results we observe that for samples grown on PET substrates at 350 W, the nanowires diameter increases when the heating time increases from 5 to 35 min according to the growth mechanism. Increasing the furnace power at 700 W, the nanowires diameter decreases in needle tip form, but their length is increased. This is in agreement with Unalan et al. [11]. For samples grown on glass substrates at 350 W, a relationship between the heating time and the nanowires diameter was not observed. At 700 W the nanowires diameter remains almost constant in 200 nm, independently of the heating time conditions. For samples grown on Si substrates at 350 W and 700 W, the interval of the nanowires diameter remains constant from 200 nm to 400 nm and from 70 nm to 200 nm, respectively. This shows that the nanowires diameter grown on Si is independent of the heating time. Both on PET and on glass it is observed that the growth of nanowires with 35 min of heating decreases the diameter of the same but increases its length. Besides, it can be observed that the samples grown on PET substrates show a better alignment of the ZnO nanowires compared to those grown on glass or Si substrates.

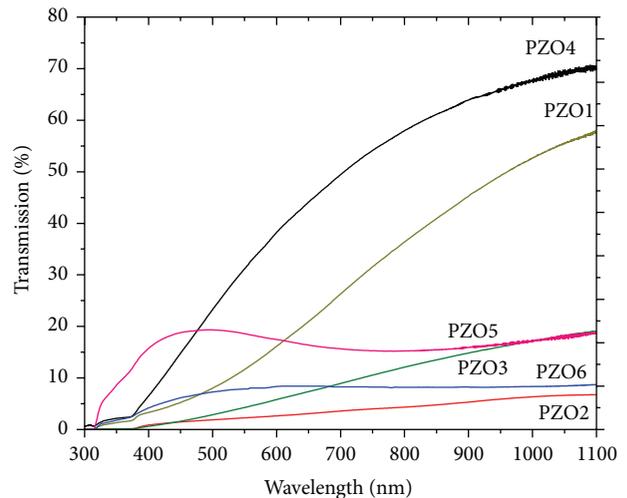


FIGURE 10: Optical transmission spectra of ZnO nanowires grown on PET substrate.

It was showed that, in PET, glass, and Si substrates, the crystal orientation changes with furnace power condition and the heating time. At 700 W and 5 min, the ZnO nanowires exhibit a preferential alignment along *c-axis* direction. Namely, the formation of highly oriented ZnO nanowires requires high energy and low heating times. While at smaller furnace power as 350 W, the strongest reflection was the (100) compared to (002) and (101), showing more isotropic and randomly oriented samples. In particular for glass substrate this behavior was observed increasing the heating time.

In a substrate area of 8 mm × 8 mm, approximately, we observed uniformity of deposition on glass and PET substrates. However, ZnO nanowires on Si were grown as islands dispersed over that area.

**3.3. Optical Transmission.** The optical transmission spectra in the range of 300 nm and 1100 nm for samples grown on PET and glass substrates at room temperature are shown in Figures 10 and 11, respectively.

The optical transmission spectrum for sample PZO4 (Figure 10), which was the thinnest film according to X-ray diffraction patterns, shows the highest transmission, contrary to sample PZO2 which was the thickest film showing the lower transmission. From the transmission spectra the band gap energy was obtained from each sample. It was not possible to determine the band gap energy of the sample PZO5 through this method. In general, it can be observed that there is not so much variation in the value of the band gap energy, which remains constant independently of the conditions of the growth parameters. Sample PZO4, which presents the higher transmission of around 70%, showed the best value of the band gap energy which is the closest to bulk ZnO of 3.37 eV.

The optical transmission spectra for samples GZO4 and GZO5 grown on glass substrate (Figure 11) show good transparency in the visible range, which were the thinnest films according to X-ray diffraction patterns. From the

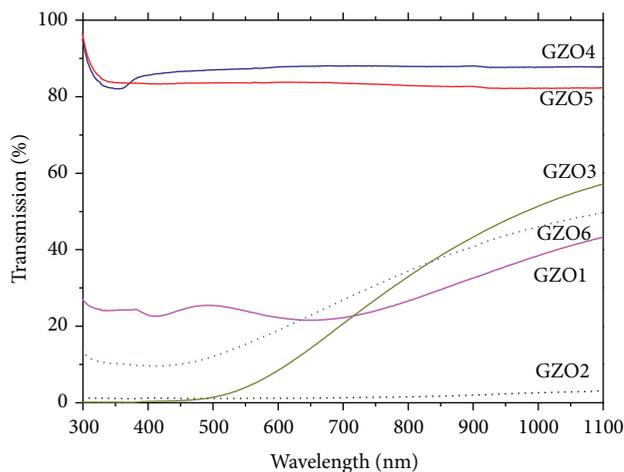


FIGURE 11: Optical transmission spectra of ZnO nanowires grown on glass substrate.

transmission spectra the energy gap  $E_g$  was obtained from each sample (Table 1). It was not possible to determine the energy band gap of the sample GZO5 through this method. Sample GZO4 with band gap energy of 3.33 eV and with a transmission of around 88% shows the closest value to bulk ZnO of 3.37 eV. XRD patterns show that when the heating time is 5 min there is a preferential growing of ZnO at 350 W, which is observed by the reflection at  $31.64^\circ$  corresponding to the direction (100), and for 700 W it is observed by the reflection at  $34.27^\circ$  (002). On the other hand, when the heating time is 35 min there is not a preferential growth of ZnO that in both conditions of 350 W and 700 W is observed by the reflections at  $31.64^\circ$ ,  $34.27^\circ$ , and  $36.16^\circ$  (101). These results are in agreement with that observed in the SEM images.

#### 4. Conclusions

ZnO nanowires were grown on several substrates (PET, glass, and Si) by the hydrothermal method using a rapid microwave heating process. It was possible to vary the diameters of the ZnO nanowires altering the power and heating time for achieving ZnO nanowires with diameters smaller than 50 nm. Using the same growth parameters on the three substrates, it can be observed that in high power (700 W) and varying the heating time of 5 to 35 min the growth of ZnO nanowires on PET is favored, but heating conditions, as 700 W and 5 min, promote the preferential orientation in  $c$ -axis directions in all the three substrates. On PET substrate at 700 W of furnace power, a correlation between the heating time and the diameter of nanowires was observed. For larger heating time, smaller diameter and larger length are obtained in the nanowires. For heating time of 5 min, the nanowires grow aligned perpendicular to the substrate. At 700 W of furnace power, the growing of ZnO nanowires on PET, glass, and Si substrates showed a correlation between the heating time and the length of nanowires. For larger heating time, larger length is obtained. The value of the band gap energy obtained

for the samples grown on PET and glass remains almost constant independently of the growth parameters.

#### Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

#### Acknowledgment

This work was supported by SIP, Project 20144394, Mexico.

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