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EFFECT OF DEPOSITION TIME OF ZNO NANORODS BY HYDROTHERMAL METHOD ON PHOTOCATALYSIS ACTIVITY

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Abstract: *Zinc oxide Nanorods are grown by hydrothermal method on seed layer of ZnO for various deposition times at low temperature. The seed layers were deposited on soda lime glass by RF Sputtering with 100 nm of thickness. This study aimed to investigate the influence of deposition time of ZnO Nanorods on the photodegradation of Methylene Blue. It is well known that the experiment conditions control the growth of ZnO NRs. The crystalline structure of ZnO NRs was studied by means of X-ray diffraction and shows that the ZnO NRs obtained are primally well aligned and perpendicular to the substrate. Moreover, ZnO nanorods can be grown with a strong orientation along (101) and exhibit a wurtzite crystal structure in an XRD spectrum and possessed a high crystal quality. İn addition the strong relative intensity of the (002) lines reveals a texture effect of the arrays consistent with c-axis oriented nanorods. The Photocatalysis activity has more efficiency when the deposition times decrease.*

Key words*: Zinc oxide, Hydrothermal method, Deposition time, Nanorods, RF Sputtering*

1. Introduction

Nanoscale materials have unique mechanical, electrical and thermal properties that make them very attractive in research and health related applications. Among these, one dimensional (1D) Zinc oxide (ZnO) nanorods (NRs) is a multilateral semiconductor which has been intensely studied as a promoting materiel for many industrial applications. Zinc oxide, a II–IV semiconductor, has a wide direct gap of 3.37 eV at room temperature and large exciton binding energy of 60 meV [1],has attracted considerable attention because of their application potential in a variety of fields, including chemical sensors, photo detectors, field-effect transistors (TFTs), piezoelectric generators, solar cells [2-7] and photocatalysis activity [8],[9].

Various synthesis methods have been reported for 1D ZnO nanomaterials growth [10–14]. However, these methods always require a catalyst or severe conditions for nanowires (NW) growth.

In this study, we report that the vertical well-aligned ZnO NRs arrays were prepared on ZnO/soda lime glass templates by hydrothermal method, where ZnO seed layers were grown on glass via radiofrequency sputter (RF-sputter) deposition with thickness of 100 nm. The effects of deposition time on the growth of ZnO NRs have been investigated in detail. As a result, the well-controlled method on the morphology of ZnO nanorods arrays can be utilized in the improvement of the carrier collection for hybrid polymer photocatalyst devices based on ZnO.

2. Experimental

Before NRs synthesis by hydrothermal, ZnO seed layers with thicknesses of 100 nm were deposited onto soda lime glass substrates using radio frequency (RF) magnetron sputter-deposition technique. A glass substrate was placed on the top of an anode, which was placed 50 mm from the ZnO target of 3 in. diameter. During the growth, the working pressure of the chamber was 10 m Torr while the RF power was 40 W and the Ar gas flow was maintained at 5, 5 sccm. No substrate heating and bias were applied during the deposition.

The chemicals used in this work, zinc nitrate hexahydrate [Zn $(NO_3)2$ $6H_2O$, 98%] and sodium hydroxide [NaOH] were purchased from Aldrich and used as received. The growth solution was prepared by dissolving a calculated amount of zinc nitrate hexahydrate and sodium hydroxide in deionizer (DI) water. Successively, the addition of the hydroxide sodium is to adjust the pH of the growth solution. Then the glass substrates with ZnO seed layers were immersed in the growth solution in Teflon Lined Stainless Steel Autoclave and put them in oven at 120°C for 3 h, 5 h and 8 h. Finally, the ZnO NRs samples were removed from the solution after cooling, rinsed with distilled water and dried in air at 60°C.

The X-ray diffraction (XRD) was used to study the crystallographic properties. The optical properties of the obtained ZnO NRs were characterized by UV-Visible spectroscopy. The photocatalysis activity was investigated by the photodegradation of Mythelene Blue with various time depositions of ZnO NRs.

3. Results and Discussion

Fig 1 shows XRD spectra of ZnO NRs prepared by hydrothermal at 120°C with different deposition time (Fig 1.a) deposited with 5 H and (Fig 1.b) with 8 H . As can be seen, the both samples are polycrystalline in nature with of the crystal growth along the plane (101) having a hexagonal wurtzite structure. Moreover the ZnO NRs with 8 H have more intensities than the previous one. It could be seen that the diffraction peaks were more intensive and narrower implying a good crystalline nature of the as-synthesized ZnO NRs, and all of the peaks can be well indexed to hexagonal phase ZnO reported in JCPDS card (NO.36-1451, a = 0.3249 nm, c = 0.5206 nm). Diffraction peaks related to the impurities were not observed in the XRD pattern, confirming the high purity of the synthesized NRs. These results are in a good agreement with Sini K. et al [9].

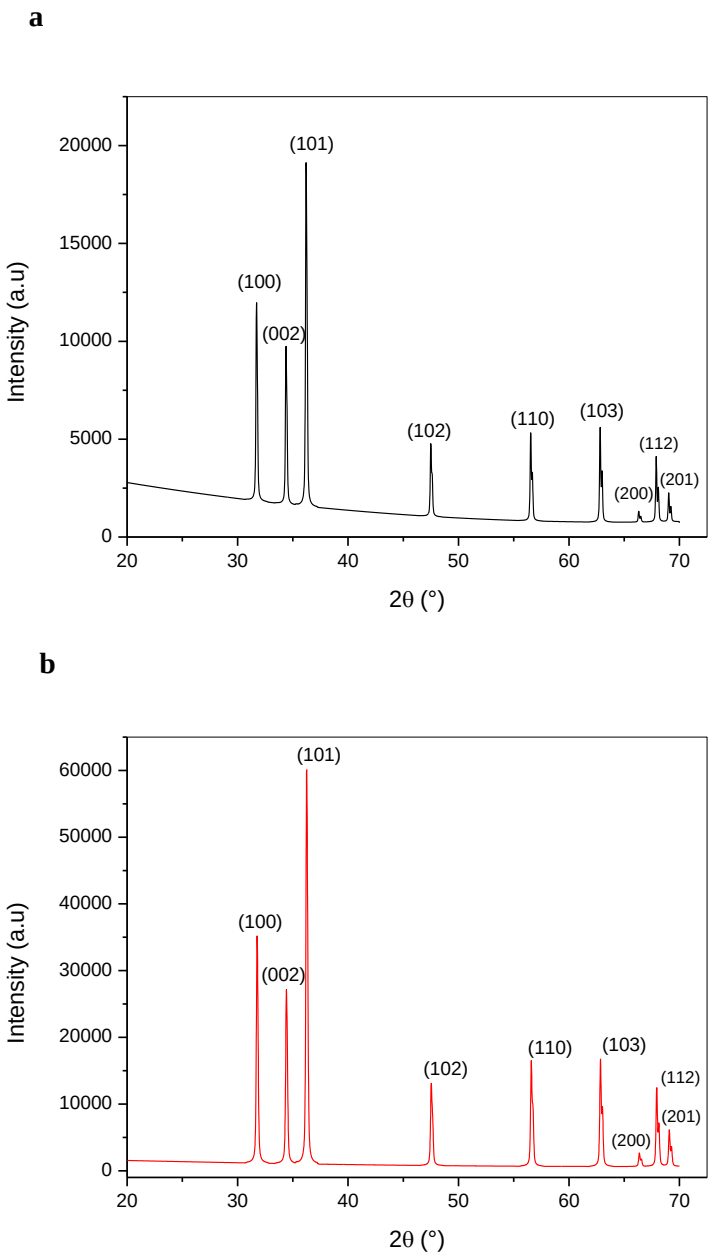

Fig. 1: XRD patterns for ZnO Nanorods with different deposition time: (a) 5 h and (b) 8 h

The samples prepared were used to research for treatment of water sush as the photocatalysis activity. The photocatalysis activity was studied by Methylene Blue as a polluant with a concentration of 10^{-5} mol/l and the lamp used in this study is a lamp with a wavelength of 254 nm.

We have studied the variation of absorbance spectra of polluted solution after UV light exposure times. The wavelength region is ranged from 500 to 700 nm; this broad absorption band is a characteristic of the used Methylene Blue (MB) dye. The absorption peak intensity is used as signature of the dye degradation. From the variation of this absorption peak with exposure time, we concluded

that the photo degradation is more significant when solution is in contact with ZnO Nanorods which deposited for film tested with 3 H as time deposition.

To have more insight on the photo degradation kinetics and the influence of deposition times of Zinc Oxide Nanorods, we have monitored the variation of the intensity of the absorption located at 664 nm.

Fig.2 exhibits the variation of the ratio C/C_0. As shown, the photo degradation is more efficiency after 60 min when using 5 hours as a deposition time for ZnO Nanorods. The ratio is reduced with increasing exposure time; it reaches 0.35 after 240 minutes of irradiation for film with 3 h. However in the case of the other films the ratio is slightly increased up to 1.03 and 1.45 after 240 minutes for films deposited for 5 h and 8 h respectively.

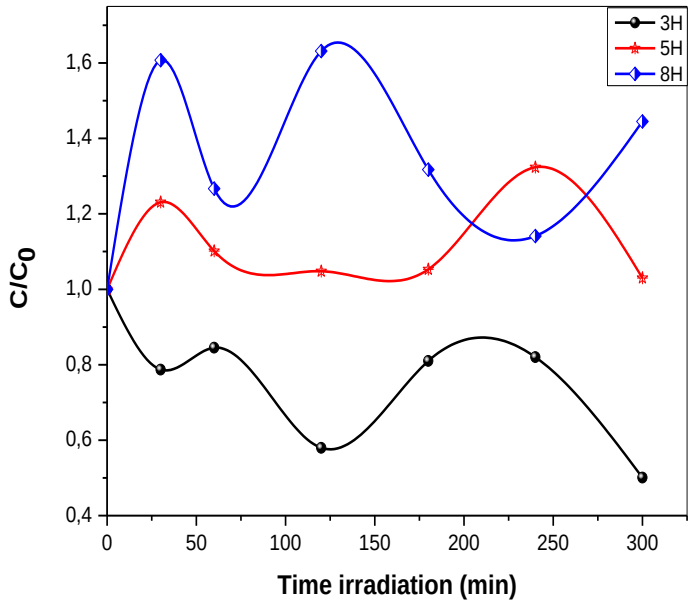

Fig.2: Photo degradation Kinetic of Methylene Blue by ZnO NRs with different deposition time

As can be seen in Fig.3, the photocatalytic decomposition of MB pollutant, in contact with the surface of ZnO Nanorods with different deposition times, follow a pseudo first-order kinetic law, it can be expressed as:

$$Ln\left(\frac{C_0}{C}\right) = kt \qquad (1)$$

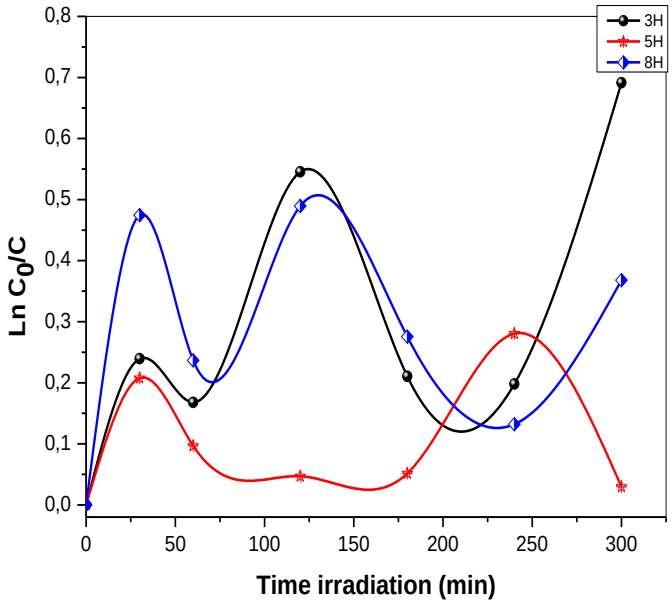

Fig. 3: Ln (C_0/C) of Methylene Blue by ZnO Nanorods with various deposition times used a lamp with 254 nm

In order to understanding better the results obtained, we calculated the conversion rate (τ) of the photodegradation of the BM by the set of samples. This ratio represents the ratio between the amount of transformed reagent and the initial quantity, using the following equation:

$$\tau = \frac{C_0 - C_t}{C_0} * 100 \qquad (2)$$

Fig.4 shows the time evolution of the conversion of Methylene Blue for all the experiments, calculated using equation (2).

As can be seen 22% of MB is removed after 60 min of exposure time by ZnO NRs deposited for 3 H then after 240 min the conversion rate is reduce to 30%. While in the second case and after 60 min of irradiation time only less than 19% is removed when ZnO NRs deposited after 5h. Then the photodegradation is more important when the film is in contact with the solution under irradiation after 240 min the ratio is equal to 50%.

For the third samples ZnO NRs obtained after 8 h the ratio is reached 29% after 60 min but we observed the increase of this ratio until 45 % after 240 min of irradiation times.

According to this survey, the photodegradation of M.B is more significant when the samples which deposited after 5h are on contact with solution and the sample with 3h of deposition time has a larger constant of reaction K then the other samples.

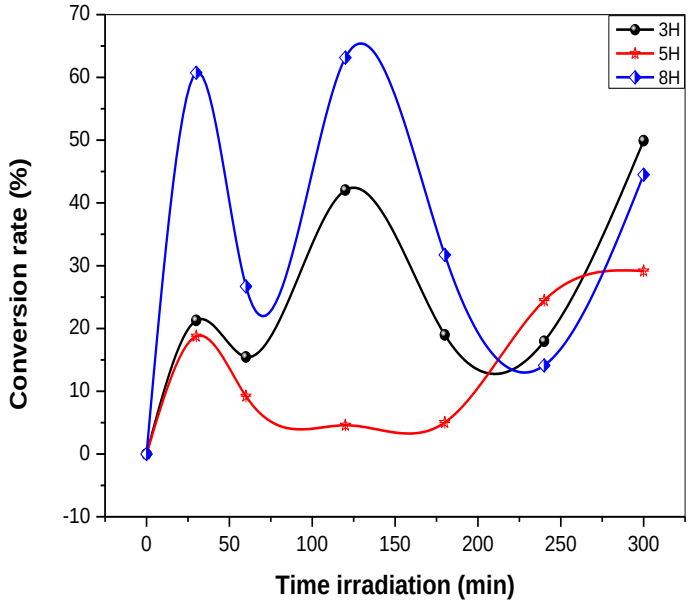

Fig. 4: Conversion rate of Methylene Blue by ZnO NRs with various deposition times

In Tab.1 we regrouped the values of the conversion rate and the constant of reaction K which are give us an idea about the photodegradation of the polluant Methylene Blue by ZnO Nanorods obtained by hydrothermal at 120°C with several deposition times 3, 5 and 8 h.

ZnO Nanorods deposited with	Conversion rate τ (%)	Constant of reaction K (min^{-1})* 10^{-4}
3 H	30	13.6
5 H	50	1.12
8 H	45	2.16

Tab.1: the values of all parameters of the photocatalysis activity

4. Conclusion

In this present study we demonstrate that the growth of ZnO Nanorods can be performed by a simple and low cost method of growth from precursors in solution on ZnO seeds deposited by RF sputtering on soda lime glass substrate. Such materials could be suitable for solar active devices such as dye-sensitized solar cells or photocatalytical devices for water decontamination. Although growth conditions influences the properties of the final material, the composition of seeding solution –in particular, the solvent- is the relevant parameter for controlling the structural and optical properties. The technique used in this work is a novel low temperature, and thus low cost method that could be extended to other metal oxide nanostructured materials.

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