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Effect of Yttrium Doping on the Photocatalytic Properties of ZnO Thin Films

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Zinc oxide films with different levels of yttrium doping are deposited on glass substrates, using radio-frequency magnetron sputtering. Photocatalytic properties were investigated for such Y-doping weight concentration: 0, 2.4, 3.9, 4.7 %. The studies showed that the Y-doping significantly improves the photocatalytic activity of the ZnO thin films. It was shown that the ZnO:Y 3.9 wt. % presents the highest degradation efficiency of 100 % during 80 minutes and the largest rate constant $9.6 \cdot 10^{-2}$ min^{-1} among all samples.

Key words: zinc oxide, photocatalysis, photodegradation, absorption spectra.

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Introduction

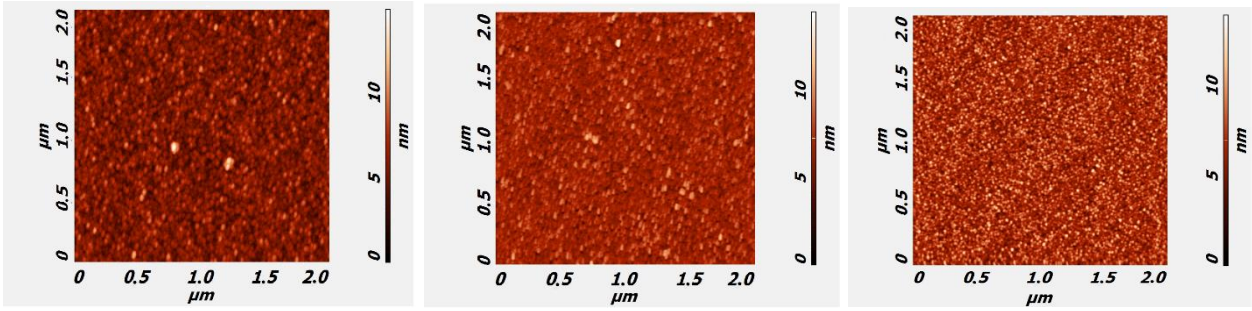
Major problem of whole world is water contamination, due to several reasons like inadequate sewage treatment, industrial wastes, marine dumping issues, radioactive waste material, some agricultural pesticides etc. [1]. Water pollution has an adverse effect on environment, and it can also be responsible for air pollution that reflects very dangerous results on human health.

Photocatalysis provides a potential offer to purify water with a low expense, high working efficiency in removing pollutants and reusable ability. In recent years, increasing attention among semiconductor materials in heterogeneous photocatalysis has been paid to ZnO [2]. In addition, it was confirmed the enhancement in photocatalytic degradation by using metallic ions-doped-ZnO. There are a lot of numbers of publications about different metallic ions-doped-ZnO [3-11]. But despite great potential practical and fundamental interest, the number of publications that deal with the photocatalytic properties of yttrium-doped zinc oxide films still remains insufficient [12-17]. Moreover, we didn't find publications, which describe photocatalytic properties of yttrium doped ZnO thin films synthesized by radio-frequency (RF) magnetron sputtering technique. The RF magnetron sputtering is among the best techniques applied when manufacturing thin oxide films. Its advantages include suitability for sputtering of highly melting materials, versatility and low cost. The efficiency of the RF magnetron sputtering is owing to high activity of gas component molecules, which is stimulated by the action of RF plasma [18]. An important feature of the sputtering process is that the chemical composition of a sputtered film will often be the same as that of the target from which it was sputtered [19].

In this report, we present the results for the influence of different levels of yttrium doping (0, 2.4, 3.7, 4.7 wt. %) on photocatalytic properties of ZnO thin films. Our films are deposited on glass substrates using the RF magnetron sputtering technique.

I. Materials and methods

Zinc oxide films with different levels of yttrium doping are deposited on glass substrates, using RF magnetron sputtering in argon atmosphere with working gas pressure 0.1 Pa, high-frequency oscillator power 75 W, distances between target and substrate 60 mm, magnetic field induction 0.1 T, and substrate temperature of 300°C



a) b) c)

Fig. 1. Three-dimensional atomic force microscope micrographs obtained for ZnO:Y films with different yttrium concentrations: (a) 2.4, (b) 3.9 and (c) 4.7 wt. %.

The target was prepared from a compressed mixture of ZnO (chemically pure grade) and Y_2O_3 (pure grade) powders, used in appropriate proportions [18]. The sputtering time was 1 hour. According to ellipsometric data, the thickness of the films was about 0.6 μm. The surface morphology of our samples was studied using a Solver P47-PRO atomic force microscope.

ex situ ellipsometry measurements were performed with a serial null-ellipsometer LEF-3 M in a standard PCSA (polarizer–compensator–sample–analyzer) arrangement. He–Ne laser (λ = 632.8 nm) was a light source.

For photocatalytic application, methyl orange ($C_{14}H_{14}N_3NaO_3S$) was selected as the organic dye for testing the photocatalytic properties of all Y-doped ZnO samples. The studied sample in the form of a plate with overall dimensions of 4.1 mm × 6 mm × 1 mm was placed into a standard 3.5 mL quartz cuvette with aqueous solution of methyl orange (22 mg/L). Prior to illumination, the sample was immersed in the methyl orange (MO) solution for about 20 h in darkness in order to achieve an adsorption desorption equilibrium state. Then, the MO solution containing the sample was irradiated cyclically every 20 min over a period of 2 h by a DRT−125 Hg-quartz lamp (with the power of the UVVIS source 125 W, among them ultraviolet was 70 W; with wavelengths of the irradiation 220 - 400 nm and light ow 1850 lm). The desired irradiation intensity can be achieved by the variation of a distance from the lamp. In this work, the cuvette containing MO solution with a sample was placed at the distance of 5 cm from a source of light.

To evaluate the photocatalytic efficiency, the optical density at the dye absorption maximum (465 nm) was measured using a portable fiber optic spectrometer AvaSpec-ULS2048L-USB2-UA-RS (Avantes BV, Apeldoorn, Netherlands). This spectrometer was also used to study the absorption edges of ZnO:Y films. The detection of light in the spectrometer was carried out by a 2048 pixel CCD detector. The special software for automate computer control for this type of spectrometer and spectra processing was used (AvaSoft 8, Apeldoorn, Netherlands).

II. Results and discussions

Surface analysis of the prepared undoped and yttrium doped ZnO films were performed with the atomic force microscope. Figure 1 shows the AFM images in scale 2×2 μm^2, which were used for obtaining root mean square roughness and average grain size of each doped sample. As can be seen in Table 1, the tendency to decrease the average size of crystallites with increased yttrium concentration was observed.

Table 1.

Root mean square roughness, average grain size and range of sizes for ZnO:Y films as functions of yttrium concentration

Yttrium dopant concentration, wt.%	Root Mean Square roughness, nm	Average grain size, nm
2.4	1.6	6.9
3.9	0.8	6.7
4.7	1.3	5.7

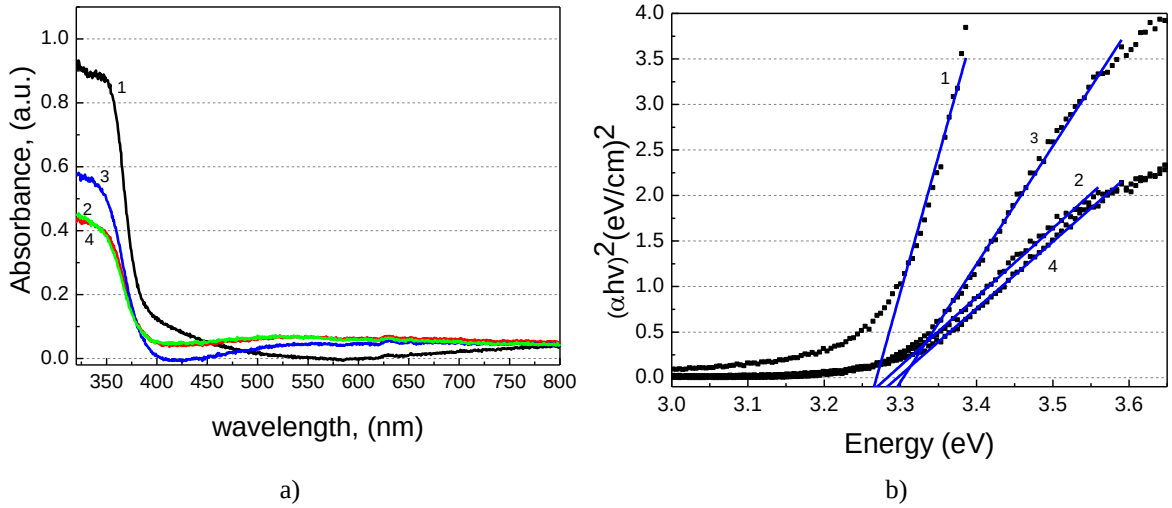
Although a few larger grains are visible in the images (b, c), their number is not significant and does not affect the overall distribution and roughness. But with a higher concentration of Y, according to the literature [18, 20-22], the number of larger grains becomes significant, and then the average grain size and roughness increase, respectively.

Studying the optical band gap of the sample is expected to help deeper understand of the photocatalytic properties. Fig. 2a depicts the room-temperature absorption spectra of undoped and Y-doped ZnO films. It can be observed that the yttrium doping influences the optical behavior of the films considerably. The absorption edge of the ZnO films after doping shifts to smaller wavelengths of light. The transparency in the visible spectral range is maximal for pure ZnO film and it slightly decreases with incorporation of the Y. These results are in good agreement with the data reported in [21, 23].

To estimate the optical band gap (E_g), the Tauc's relation was plotted as shown in Fig. 2b, according to Eq. (1) [18].

$$ahv = B\left(hv - E_g\right)^r , \quad (1)$$

where a is absorption coefficient, hv – the photon energy, B is a constant that remains practically independent of the photon energy and r can assign values of 1/2, 2, 3/2, and 3 respectively corresponding to the allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions.



a) b)

Fig. 2. Room-temperature absorption spectra of ZnO films with different doping level (*1*: 0 wt. %; *2*: 2.4 wt. %; *3*: 3.9 wt. %; *4*: 4.7 wt. %) plotted a) in the coordinates α vs wavelength and b) in the coordinates (αhν) vs. *hν*.

In our case we deal with direct allowed transitions (r = 1/2). Plots of $(\alpha h\nu)^2$ versus $h\nu$ (Fig. 2b) otherwise known as Tauc's plots are used to calculate the band gap of the photocatalysts. E_g is obtained as the plot tangent intercept at the X-axis. The optical band gap values for the undoped ZnO, 2.4 wt %, 3.9 wt % and 4.7 wt % Y-doped ZnO, were 3.275 eV, 3.28 eV, 3.295 eV and 3.28 eV, respectively. Increasing band gap up to 3.9 % can be explained by the Burstein–Moss effect. In other words, band gap energy shows a blue shift with increasing Y-ion concentration similar to the Fermi level shifts with increase in the carrier concentration, which blocks the lowest conduction band levels and increases the band [15]. Above 3.9 wt% the optical band gap begins to decrease is due to 'apparition' of energy states in the forbidden band close to the band edges [18].

The effect of yttrium doping was studied by conducting a series of experiments with different amounts of Y (0, 2.4, 3.9 and 4.7 wt. %) with constant parameters of the photocatalytic experiment. Figure 3 (a-d) shows the photocatalytic degradation of MO in 100 minutes by using the photocatalyst of films of ZnO and ZnO:Y (with 2.4, 3.9 and 4.7 wt. %).

Fig. 4a shows the change in concentration vs. irradiation time in the absence and presence of different photocatalysts. The decolorization efficiency of an aqueous solution of MO can be calculated by using the following relationship [12]:

$$Ef_{Dec} = \frac{C_0 - C_t}{C_0} \times 100\% \ , \qquad (2)$$

where C_t is the peak intensity ratio calculated for the different samples after adding MO and exposed to irradiation for 20 min and C_0 is set at 100 for the reference peak intensity measured for the MO solution without any irradiation.

The efficiency of dye decomposition was 73.3, 92.8, 99.8 and 88.5 % for ZnO films samples with dopant concentrations 0, 2.4, 3.9 and 4.7 wt. %, respectively, for 60 minutes UV irradiation (Fig. 4b). As can be seen, the photocatalytic efficiency of the Y-doped ZnO photocatalysts increases with an increase in Y concentration from 2.4 wt. % to 3.9 wt. %, a further increase in Y concentration led to a decrease in the efficiency of the photocatalyst, which also confirms in the paper [13].

These results are consistent with our absorption edge data, as the photocatalytic properties improve as long as the Berstein–Moss effect, i.e., the optical band gap increases. In addition, according to the article [14] yttrium plays a significant role to decrease particle size thus leading to enhanced photocatalytic performance. As was shown above, decrease average grain size was observed in obtained doped ZnO films.

Using the graphical method it was observed that photodegradation of methyl orange by ZnO and ZnO:Y films followed first-order kinetics [24]. Figure 5 depict the kinetics of MO degradation under UV catalytic activities by different photocatalysts. The value of reaction rate constant (k) for ZnO:Y films with dopant concentration 0, 2.4, 3.9 and 4.7 wt. % is found to be $2.5 \cdot 10^{-2}$ min^{-1}, $3.5 \cdot 10^{-2}$ min^{-1}, $9.6 \cdot 10^{-2}$ min^{-1} and $4.3 \cdot 10^{-2}$ min^{-1}, respectively. A bar-chart (Fig. 5.b) was constructed to visualize the difference between the reaction constants of different doping concentrations.

It was observed that Y-doping of ZnO films would have a positive effect on photocatalytic activity of obtained films. The reaction rate constant for ZnO:Y with 3.9 wt. % of yttrium increased more than three times compared to the undoped sample. In [14] Y-doped ZnO with 4 mol. % yttrium doping presented the maximum degradation efficiency among all samples. They explained the enhancement by synergy of decreased particle size and reduced E_g. In our samples also observed decreased average grain size but the best result had the largest value of E_g. Such differences in the photocatalytic mechanism could be associated with a completely different method of synthesis (ZnO:Y films are synthesized using the RF magnetron sputtering method).

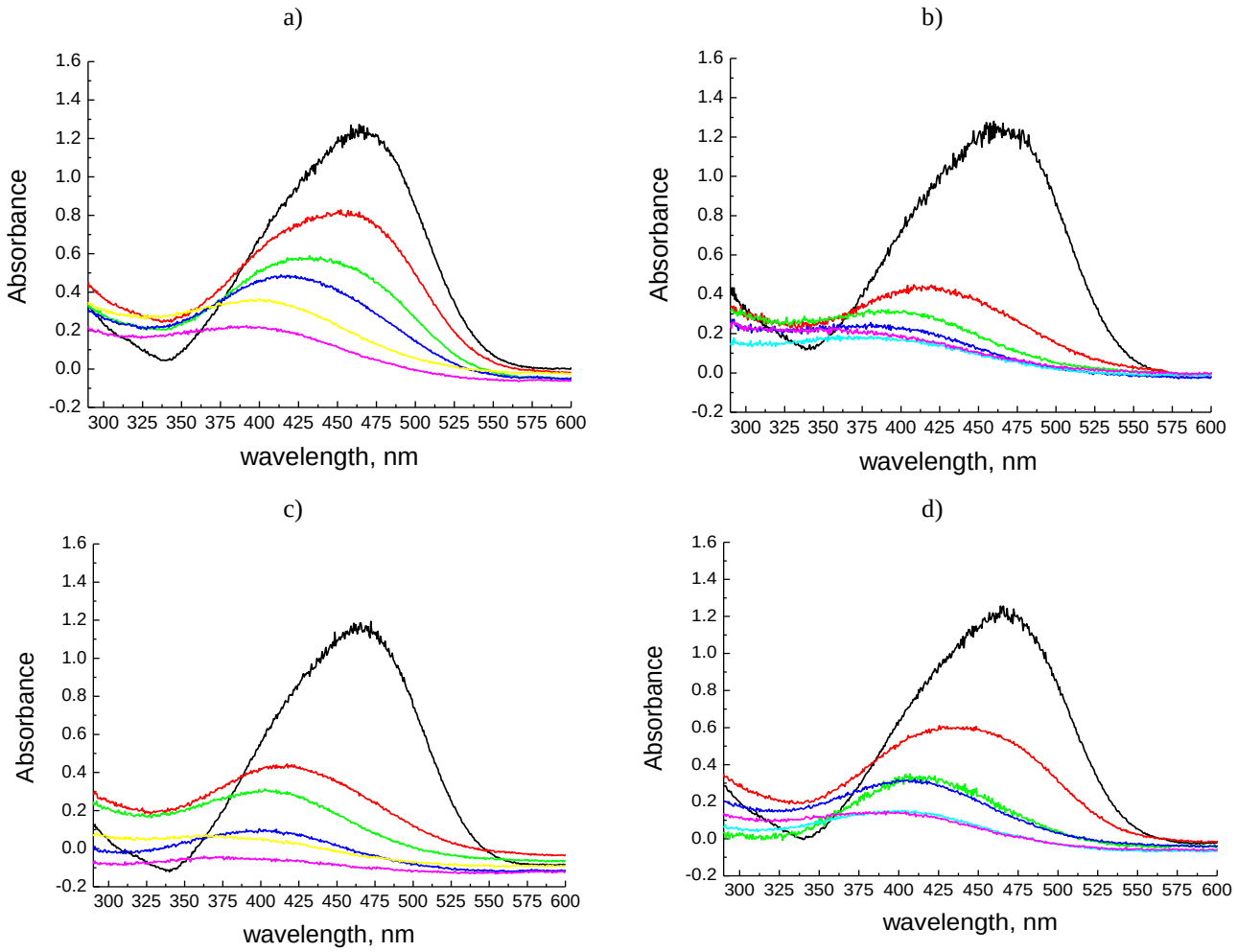


Fig. 3. Absorption spectra of an aqueous solution of MO which were taken every 20 minutes of irradiation with a DRT-125 lamp for two hours by using ZnO films with different levels of yttrium doping: (a) undoped, (b) 2.4, (c) 3.9 and (d) 4.7 wt. %.

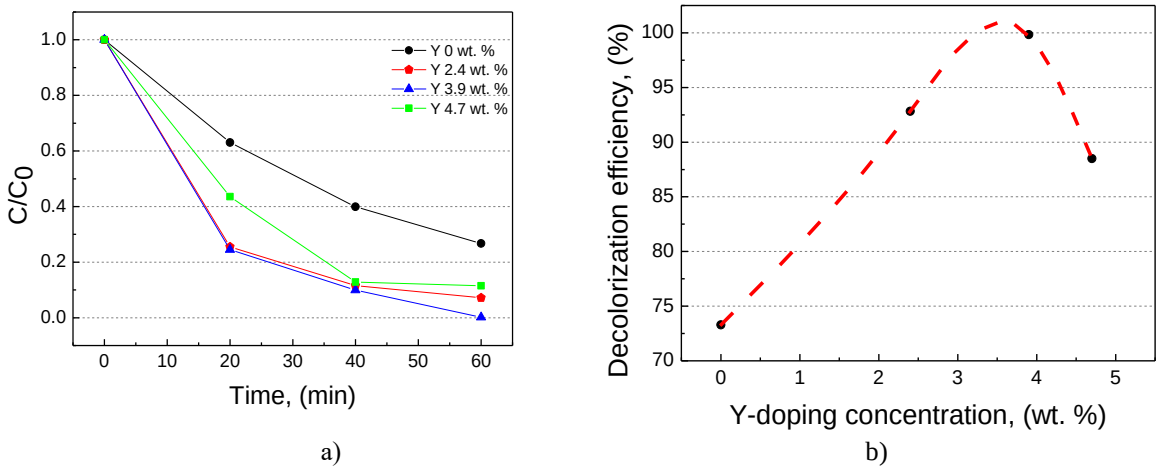
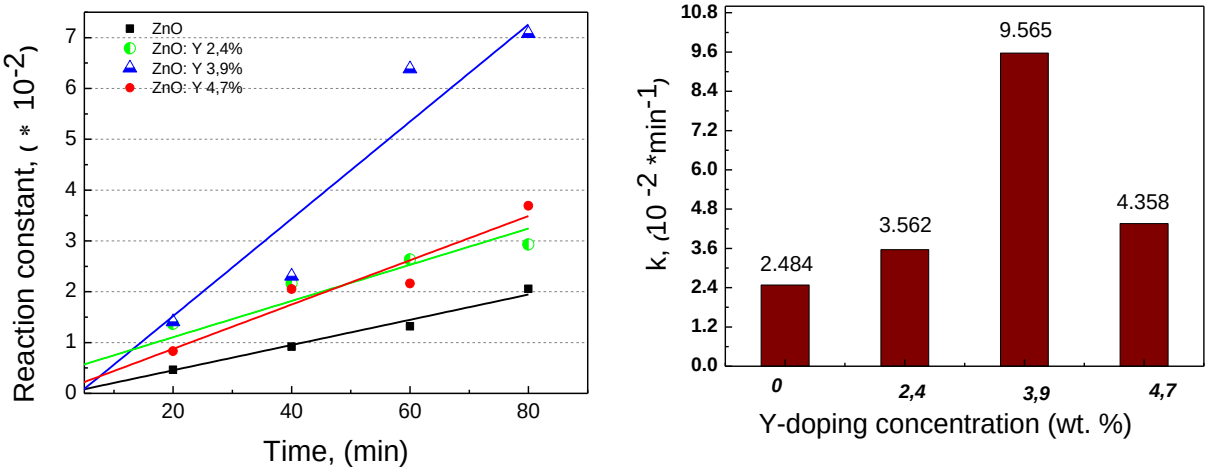


Fig. 4. The change in concentration vs. irradiation time a) and degradation efficiency of MO using different photocatalysts b). Cubic spline method is used for degradation efficiency interpolation.

Parangusan et al [15] reported the best photocatalytic properties are possessed by the ZnO:Y 12 % for which the value of the blue shift is the largest. Authors concluded that the Y-doping leads to a blue shift in the emission bands and increase in the surface defects and oxygen vacancies. The results of X-ray diffraction studies for characterization of zinc oxide films with different levels of doping with yttrium were presented by us in our previous work [18]. For all our experimental samples, the peak (002) was only one in the X-ray diffraction spectra. For any experimental film samples, no diffraction peaks associated with presence of the metallic yttrium or its oxides were found. In [25] peek (002) of synthesized ZnO films, which were obtained by the RF magnetron



a) b)

Fig. 5. Decolorization kinetics of methyl orange using ZnO and Y-doped ZnO photocatalysts a) and representation of these reaction constants in the form of a bar-chart b).

sputtering method with P lower than 200 W, characterized the presence of a substantial amount of hydroxyl group attached to the Zn lattice. During the photocatalytic process in ZnO, hydroxyl groups on the surface take an active part in the oxidation of organic compounds by capturing holes (h^+) from the semiconductor, which leads to the formation of hydroxyl radicals. An increase in the content of hydroxide groups on the surface increases the photocatalytic activity [26]. Based on the results and literature on this point, a possible mechanism of photodegradation enhancement might be related to decreasing average grain size and the synergistic effect between Y and defects of surface in the ZnO film catalyst has been proposed.

Conclusions

Zinc oxide films with different levels of yttrium doping are deposited on glass substrates, using radio-frequency magnetron sputtering. The effect of yttrium doping was studied by conducting a series of experiments with different amounts of Y (0, 2.4, 3.9 and 4.7 wt. %) with constant parameters of the photocatalytic experiment. Surface analysis performed with the atomic force microscope demonstrated decreased particle sizes of obtained ZnO:Y with increasing levels of Y doping. It was observed blue shift for increasing doping level from 0 to 3.9 wt. % and red shift for 4.7 wt. %. Blue shift in the optical band gap has a significant role in the enhanced photocatalytic activity.

It was shown that Y-doping of ZnO films would have the positive effect on photocatalytic activity. The photocatalytic efficiency of the Y-doped ZnO photocatalysts increases with an increase in Y concentration from 0 to 3.9 wt. %, a further increase in Y concentration led to a decrease in the efficiency of the photocatalyst. The efficiency of dye decomposition was 73.3, 92.8, 99.8 and 88.5 % for ZnO films samples with dopant concentrations 0, 2.4, 3.9 and 4.7 wt. %, respectively, for 60 minutes UV irradiation. The value of reaction rate constant for ZnO:Y films with dopant concentration 0, 2.4, 3.9 and 4.7 wt. % is found to be $2.5 \cdot 10^{-2}$ min^{-1}, $3.5 \cdot 10^{-2}$ min^{-1}, $9.6 \cdot 10^{-2}$ min^{-1} and $4.3 \cdot 10^{-2}$ min^{-1}, respectively. Mechanism of photodegradation enhancement can be explained by decreasing average grain size and the synergistic effect between Y and defects of surface in the ZnO film catalyst.

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Вплив легування ітрієм на фотокаталітичні властивості плівок ZnO

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Плівки цинк оксиду з різним рівнем легування ітрієм синтезували на скляних підкладках за допомогою технології високочастотного магнетронного розпилення. Для вагових концентрацій Y: 0; 2,4; 3,9; 4,7 ваг. % були досліджені фотокаталітичні властивості плівок ZnO:Y. В результаті досліджень продемонстровано збільшення фотокаталітичної активності плівок при застосуванні легування ітрієм. Виявлено, що ZnO : Y 3,9 ваг. % демонструє найвищу ефективність розкладання – 100 % за 80 хвилин, і найбільшу константу швидкості реакції $9,6 \cdot 10^{-2}$ $хв^{-1}$ серед усіх зразків.

Ключові слова: оксид цинку, фотокаталіз, фотодеградація, спектр поглинання.