

ZOL-GEL SYNTHESIS OF THIN FILMS ZnO /SiO₂ NANOCOMPOSITE

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Abstract. Using saponin extract as a surfactant, thin films of mesoporous ZnO /SiO₂ nanocomposite were synthesized using Sol-Gel technology and deposited on glass. The surface morphology of the resulting thin films was studied by atomic force microscopy, the elemental composition by micro-X-ray diffraction analysis, and the presence of functional groups by IR spectroscopy. The textural characteristics of the film deposited on a glass substrate using the benzene vapor adsorption method were used to determine the specific surface area of the film ($650.2 \div 780.7 \text{ m}^2/\text{g}$), the average pore diameter ($6.2 \div 10.5 \text{ nm}$) and pore volume ($0.256 \div 0.309 \text{ cm}^3/\text{g}$).

Key words: mesoporosity , film, saponin, atomic force microscopy, specific surface area, porosity.

Introduction

Semiconductor thin films prepared from ZnO are known to exhibit various unique properties. Therefore, non-toxic, low-cost thin films are quickly replacing rare and expensive materials such as indium, titanium and platinum used in solar panels, semiconductor photodiodes and transistors. Thin films based on ZnO/SiO₂ are used as semiconductor materials in optoelectronics, aerospace rockets, high-temperature structures, the production of fire-resistant materials, the creation of gas-sensitive structures for air quality control, and robotics. Also, the effective properties of ZnO and SiO₂ nanoparticles, determined by their size, are of practical importance in the production of LEDs [6-8]. The manifestation of these noted unique properties is associated with the high excitonic binding energy of ZnO (about 60 eV), which causes laser effects even at low temperatures. In ZnO/ SiO₂ nanocomposite coatings , the ZnO layer acts as a transparent and conductive layer, and the SiO₂ layer serves to prevent charge recombination and improve the efficiency of the structure [9-10]. In gas-sensitive sensors, thin nanocomposite films ZnO/SiO₂ are highly sensitive to various gases. In gas-sensitive devices, ZnO

nanoparticles are used as a sensitive material, and a SiO₂ layer is used to ensure thermal stability and selectivity [11-15]. In this regard, interest in their synthesis and study of properties is growing.

The most promising application of thin mesoporous ZnO/SiO₂ films in medicine is the use for targeted drug delivery. The film has a large surface area and porous structure, which allows it to load drugs and deliver them into the body. The ZnO layer can interact with drugs or target molecules to form a complex, and the SiO₂ layer ensures its stability and biocompatibility. Research shows that mesoporous ZnO/SiO₂ thin films can effectively deliver drugs to cancer cells and enhance their therapeutic efficacy [16].

Another potential application of mesoporous ZnO/SiO₂ thin films is tissue engineering. The surface properties of thin ZnO/SiO₂ films make it possible to reduce cell adhesion and stimulate metabolic processes in them. In addition, thin films can be used to enhance tissue regeneration and reduce cell division cycle times or provide binding of extracellular matrix proteins. Research has shown that mesoporous ZnO/SiO₂ thin films can stimulate bone regeneration and improve implant integration. In general, the synthesis of thin mesoporous ZnO/SiO₂ films and their deposition on a crimson substrate, as well as the study of their various physical and chemical properties, is relevant [17-18].

experimental part

Materials and methods

When synthesizing thin mesoporous ZnO/SiO₂ films, TEOS-(C₂H₅O)₄ Si (Jinan Xinggao Chemical Technology Co., Ltd, China, purity 98.6%) was used as a source of SiO₂, and ZnO was selected for the synthesis sols Zn(CH₃COO)₂·2H₂O (purity 99.6%). Double distilled ethanol (purity 96.2%) was used as a solvent. An HCl solution was used as a catalyst for the hydrolysis process and regulation of the environment, and saponin extracted from the root of the safflower plant with methanol was used as a surfactant.

ZnO/SiO₂ thin films.

The synthesis of a mesoporous thin film of ZnO/SiO₂ using Zol-Gel technology at 30 and 80°C using a saponin extract as a surfactant and applying it to a silica substrate was carried out in the following sequence:

1. 1 ml of saponin extract in methanol was added to 50 ml of distilled water.
2. Mix alternately prepared (concentrated) solutions of Zn(CH₃COO)₂·2H₂O and TEOS in ethanol. An HCl solution was used as a hydrolysis catalyst. The molar ratio of precursors and solvent in the prepared solution was TEOS:C₂H₅OH:H₂O:HCl:saponin 1:20:10:0.15:0.05, respectively.
3. The resulting solution was applied to the surface of the glass substrate by spraying at a speed of 10 cm/min. Spraying was repeated twice to obtain a thicker film.
4. The resulting gel was dried at 105°C for 30 minutes. To crystallize the ZnO phase and release saponin from the surfactant film, it was subjected to heat treatment at 450°C for 2 hours and a mesoporous film was obtained [19].

The results obtained and their discussion.

Benzene vapor adsorption was used to study the textural characteristics of the resulting mesoporous thin films. Benzene, obtained as an adsorbate, was first purified in vacuum and its vapor pressure was frozen until the vapor pressure given in the table for pure benzene was reached. Then it was dissolved and the gases it contained were released. Before measuring the adsorption of benzene

vapor on a sample, each adsorption system was evacuated to a residual pressure of $1.33 \cdot 10^{-3}$ Pa, kept at 273 K for 6 hours, after which adsorption isotherms were obtained. The adsorption isotherm is shown in Figure 1.

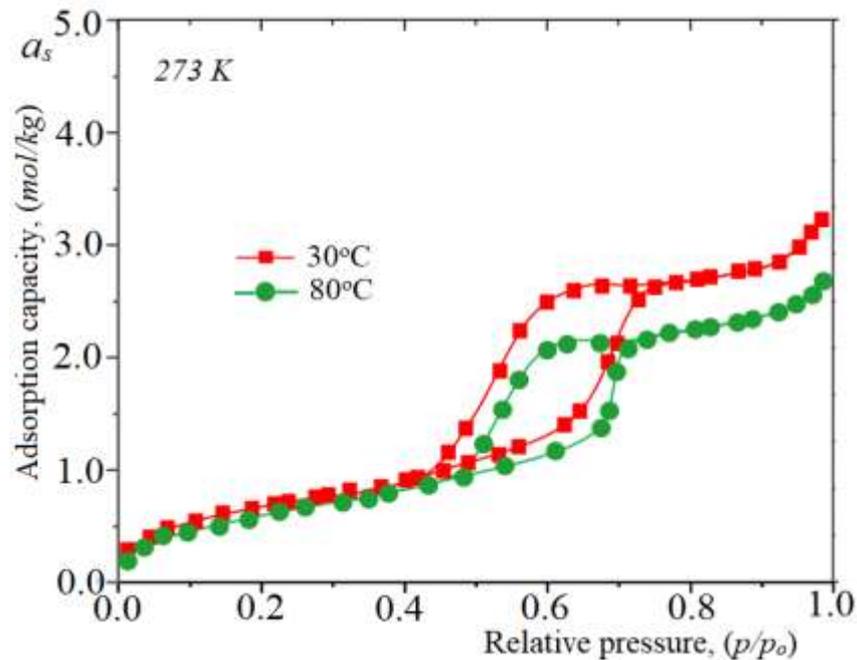
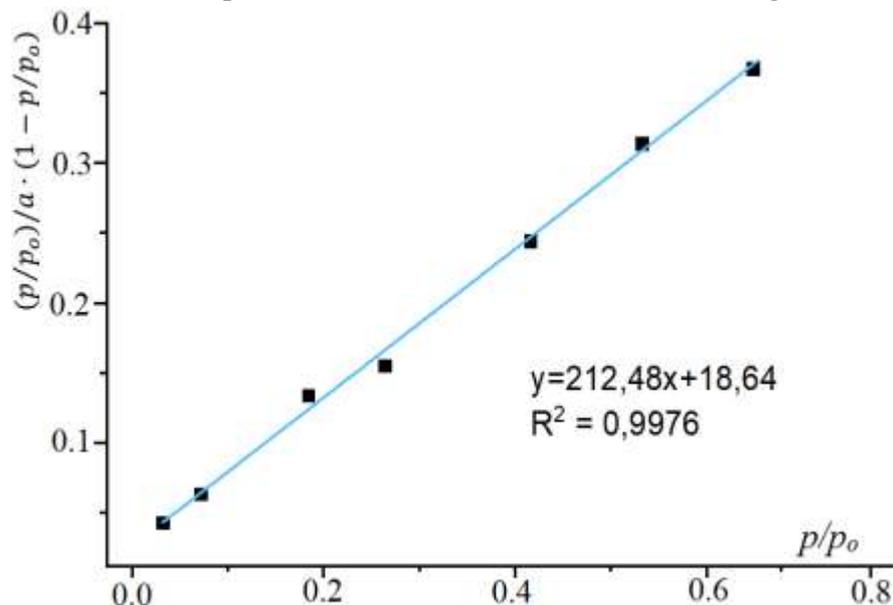


Fig . 1. Benzene vapor isotherms of mesoporous film taken at 30 and 80°C.

As can be seen from Fig. 1, the sorption of benzene vapor in films synthesized at different temperatures increased sharply up to a relative pressure $p/p_s = 0.2$ and approached the saturation state at $p/p_o = 0.9$. At $p/p_s = 0.4 \div 0.8$, it is clear that the lines of adsorption and desorption merge, forming a hysteresis loop due to capillary condensation of vapors of the adsorbed substance [20]. From this it can be noted that the resulting thin films consist of mesopores and the adsorption isotherm belongs to type IV according to the IUPAC classification. In addition, this conclusion was confirmed by the agreement of the results obtained from the adsorption isotherms with the BET isotherm (Fig. 2).



Rice. 2. Benzene vapor adsorption isotherm

From Fig . 2 shows that the isotherm corresponds to the BET model. Since the adsorption of benzene vapor in mesopores is predominantly polymolecular, the linear form of the BET equation was used to determine the monolayer capacity. According to which :

$$\frac{p/p_o}{a(1 - p/p_o)} = \frac{1}{C \cdot a_m} + \frac{C - 1}{C \cdot a_m} \cdot \frac{P}{P_s}$$

The relative surface area of the sorbent was calculated using the following formula:

$$S_m = a_m \cdot N_A \cdot \omega_m,$$

Where S_m - film surface area (m^2/g), a_m - monolayer capacity (mol/kg), N_A - Avogadro's constant , ω_m - surface occupied by one adsorbate molecule in a saturated monolayer (for a benzene molecule this value is 0.49 nm^2) [20] .

According to the BET equation, such textural characteristics as saturation adsorption (a_s), specific surface area (S_{BET}), pore volume (V_s) and their average diameter (D), as well as monolayer capacity (a_{∞}) of thin film samples at different temperatures were calculated. temperatures. The results obtained are presented in Table 1.

Table 1

Texture characteristics of films obtained at different temperatures

Sintez harorati , °C	a_m , mol/kg	S_{BET} , m^2/g	a_s , mol/kg	Dnm	V_s , sm^3/g
30°C	0.3 ±0.06	780.7 ± 10	3.5 ±0.2	6.2 ±0.8	0.4 56 ÷ 0.02
80°C	0.7 ±0.08	650.2 ±10	4.5 ±0.5	10.5 ±0.2	0.789 ÷ 0.06

How it is seen from tables , promotion temperatures during the process synthesis led to an increase average diameter pores and reduction specific surfaces _ Also was it was found that in a monolayer 8.57% of vapors are adsorbed benzene absorbed _ mesoporous film shot _ at 30°C, and 6.67% - in film removed at 80°C [20].

Topography surfaces thin mesoporous films ZnO/SiO₂ obtained at various temperatures and applied on glass , and position since then on surfaces studied with the help atomic power microscope (AEM). The image obtained in ECM and its image processed in the program FemtoScan , presented on Figure 3.

Images filmed in contact mode , on equilibrium distance of 20 nm with vibration of 5 nm . On image shown two-dimensional (2D) and three-dimensional (3D) images films 100 nm thick , processed in software ensuring FemtoScan . From the 2D image it is clear that pores on surfaces mesoporous films distributed evenly and have same size . Also By image received _ on atomic power microscope and processed in the program FemtoScan (Fig . 3b), were researched such parameters like _ thickness films and roughness surfaces _

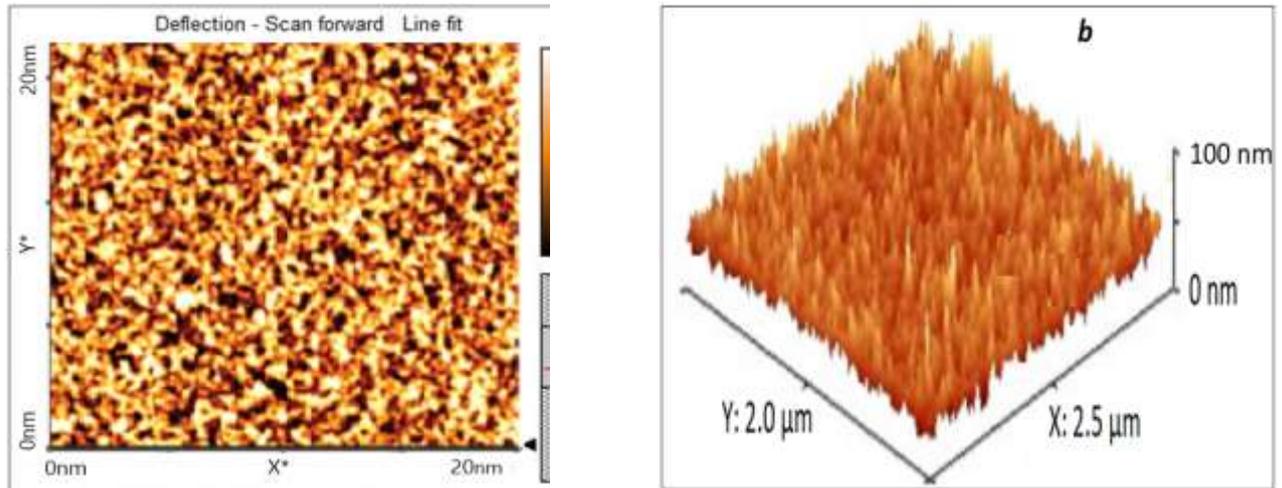
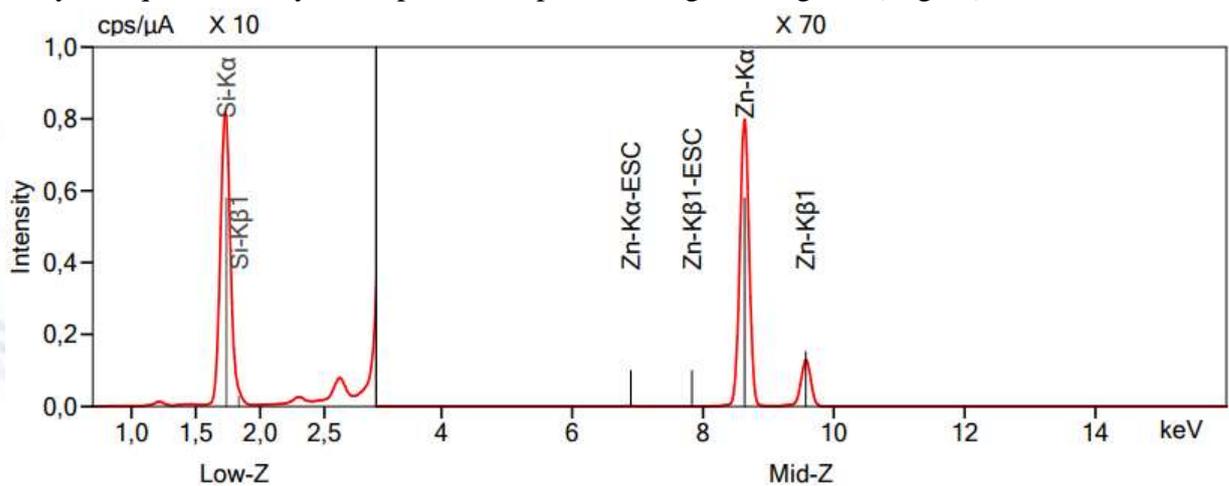


Fig.3. Images films (synthesized at 30°C), removed on Atomic power microscope and processed in the program FemtoScan .

Elemental analysis synthesized mesoporous films showed that _ her chemical compound qualitatively and quantitatively corresponds composition original reagents (Fig . 4).



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Elemental analysis established that the chemical composition of the films corresponds to 59.8% Si and 40.2% Zn by mass.

, using saponin extract as a surfactant, thin mesoporous ZnO / SiO₂ nanocomposite films were synthesized and deposited on glass using sol-gel technology . The surface morphology of the resulting thin films was studied by atomic force microscopy and the elemental composition by micro-X-ray analysis. The textural characteristics of the film deposited on a glass substrate were also studied using the benzene vapor adsorption method. Based on it, the specific surface area of the film was determined 650.2÷780.7, average pore diameter-6.2÷10.5 nm, pore volume -0.256÷0.309 cm³/g.

Conclusions

1. ZnO/SiO₂ nanocomposite films with a thickness of 100 nm were prepared at temperatures of 30°C and 80°C using sol-gel technology and deposited on a glass substrate.

2. The textural characteristics of the film deposited on a glass substrate were studied by benzene adsorption. According to it, the specific surface area of the film (UBET, m^2/g) is $650.2 \div 780.7$, the average pore diameter is $6.2 \div 10.5 nm$, and the pore volume is $0.256 \div 0.309 cm^3/g$. was full of determination.

3. The surface of mesoporous films was obtained in contact mode with vibration of 5nm at an equilibrium distance of 20 nm. It was determined that the film thickness is 100 nm, and the surface consists of pores.

4. When studying the elemental analysis of mesoporous films based on the linear intensity of the elements (sps /mA), it was found that their chemical composition corresponds to 59.8% Si and 40.2% Zn by mass.

Adabiyotlar

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