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Sensors

Optical and electrical properties of zinc oxide nanofilms deposited using the sol-gel method

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Abstract. This paper is aimed at investigation of electrical properties inherent to zinc oxide (ZnO) nanofilms prepared using the sol-gel method. The experimental samples consisted of a substrate (25×25×1 mm) made of microscopic glass brand "Voles" covered with the above films of the thickness ranging from 50 to 150 nm. Optical characterization was performed to calculate the bandgap width and to confirm the presence of zinc oxide in the nanofilms and demonstrate their optical activity. An oscillographic method was used to measure the surface resistance of nanofilms by using a galvanic elastic contact, which allowed determining their high electrical quality and resistance. Simultaneously, the high mechanical strength of these nanofilms was ascertained under the action of the elastic contact, enduring a series of 5 to 10 measurements without noticeable changes in resistance. Measurements were also carried out to observe the change in the electrical resistance of ZnO films, when they were heated from 25 up to 100 °C. The obtained results have indicated the potential of these nanofilms for applications in the fields of electronics, photoelectronics and sensor technologies.

Keywords: semiconductor zinc oxide, sol-gel method, nanofilm, transmittance spectrum, surface resistance, elastic galvanic contact.

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1. Introduction

In the modern world, nanotechnologies play a key role in the development of various fields, including electronics, optics, optoelectronics, and sensor systems. Over the past decade, nanomaterials, particularly zinc oxide (ZnO) nanofilms, have attracted special interest due to their unique electrical and optical properties [1]. Besides, ZnO is cost-effective and more environmentally friendly material as compared to many other semiconductors [2]. Zinc oxide finds a wide range of applications, from solar cells and LEDs to sensors and transistors [3–5].

However, achieving the highest efficiency in ZnO applications requires understanding their electrical properties at the nanoscale. One promising method for depositing ZnO films is the thermochemical sol-gel method [6]. The sol-gel method is a powerful tool for preparation of zinc oxide nanofilms with a high degree of control over their structure and physical properties. This method enables to achieve homogeneity and high surface quality of nanomaterials, crucial for various applications. Moreover, the sol-gel method is accessible and versatile in terms of adding doping elements during nanofilm growth [7].

Since ZnO has a bandgap of 3.36 eV, making it a wide-bandgap semiconductor, its electrical resistance is relatively high [8]. This complicates the task of investigating the electrical properties of thin nanofilms, especially when they lack doping elements that could reduce the bandgap and resistance accordingly [9].

Research of the electrical properties of semiconductor materials surface layers, such as the resistance of high-ohmic layers, requires application of specific measurement methods [10, 11]. The method using elastic contacts, a modification of measurements based on ohmic contacts, was chosen. In comparison with the often-used four-probe method [12], it allows minimizing the impact on the investigated surface and conducting measurements of high-ohmic films.

The theory of electrical contacts has been extensively presented in [13], with elastic contacts representing a specific class. Studies on this topic are covered in works such as [14]. Composite materials, including those with nanomaterials, have been recently explored for use in electronic devices as elastic contacting elements [15–18].

One promising application area for this method is pulsed voltammetry [19], characterized by the duration of measurement pulses, which can reach 100 ms or more, and pauses between pulse series exceeding 1 s. Pulsed methods for measuring the resistance of thin films and surface layers of semiconductors have recently found widespread use [20, 21].

Reducing measurement time by applying short measurement pulses (single or serial with a large interval) allows minimizing heating of thin films due to the influence of the measurement circuit on the sample. In the pulsed method, it is possible to vary both the amplitude and the time parameters of the pulses: duration, frequency of arrival and interval. This allows optimizing the measurement methodology for each type of surface – different metals and semiconductors, as well as composites and nanomaterials.

2. Materials and methods

To deposit zinc oxide to samples, which consist of a substrate (25×25×1 mm) made of microscopic glass brand "Voles", the sol-gel technology [22] was applied. For this purpose, 2.2 mg of zinc acetate were dissolved in 50 ml of isopropyl alcohol and mixed with a magnetic stirrer at the temperature of 50 to 60 °C for 30 min. As a stabilizer, 0.6 ml of monoethanolamine was added. This resulted in a colloidal sol-type solution. The obtained solution was kept at room temperature for 24 hours to form a gel.

The samples were placed in a centrifuge, and by using a pipette, the synthesized gel was deposited onto the glass surface. The samples were dried while the centrifuge rotated at the frequency 100 rpm and with simultaneous airflow at the temperature close to 70 °C [23]. The dried samples were annealed in a muffle furnace for 30 min at the temperature 300 °C to remove residual solvent, and then for an additional 1 hour at the temperature 500 °C to form the zinc oxide film. As a result, a series of samples with different numbers of layers – different thicknesses of deposited ZnO film – were obtained. Spectra and electrical resistance measurements were carried out for the samples with thickness distributions within the range 50…150 nm. For the samples with smaller thickness, a significant measurement error occurred, which was caused by the proximity to the boundary sensitivity of the applied instruments.

Optical transmission spectra of the samples were measured using the Mapada Instruments UV 1600 spectrophotometer. The measurement range was set at 300…500 nm, which is optimal for studying the samples of this type and determining the ZnO absorption threshold [24].

Sheet resistance was calculated being based on the measurement of the resistance inherent to the nanofilm area 2×1.5 mm or 3 mm². The measurement area was uniform and determined by the design of the contact module (Fig. 1). The size error limit equals the step of conductive metal threads in the rubber elastic contact: $\Delta L = \pm 0.1$ mm or ± 6%.

Elastic contacts based on anisotropic conductivity rubber "Zebra" [25], manufactured by Fujitsu, were used. Such contacts are widely used in liquid crystal displays (LCD). The elastic contacts were pressed with a clamp with a force of 32 ± 2 N, which is sufficient to ensure reliable contact [10]. The pressing area of the clamp was 3 cm². Thus, the pressure in the contact focal point equals 0.16 ± 0.01 MPa.

The contact module as part of the laboratory setup for measuring the sheet resistance of the film is shown in Fig. 1 (the clamp itself is not shown in this figure. The force is schematically indicated by F). The structural diagram of the measurement setup is shown in Fig. 2.

The laboratory setup based on a digital oscilloscope allowed for the measurement of the current flowing through the nanofilm under the influence of measurement pulses or constant voltage.

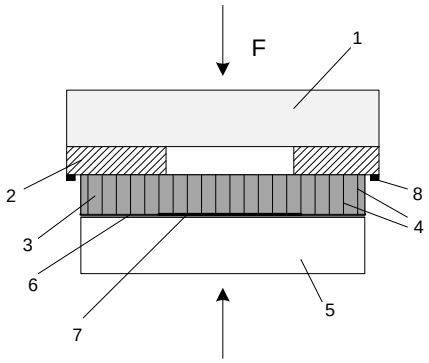

Fig. 1. Contact module of the laboratory setup for measuring the sheet resistance of the ZnO nanofilm: F – clamping force of the clamp, *1* – phenolic laminate board, *2* – lamella with Au-coating and contacts with a pitch of 1.5 mm, *3* – rubber with anisotropic-conductive elastic contacts, *4* – metal contact elements (threads), *5* – glass substrate, *6* – ZnO nanolayer, *7* – focal point of the ZnO nanolayer, where the resistance is measured, *8* – galvanic contact for mounting the signal wire.

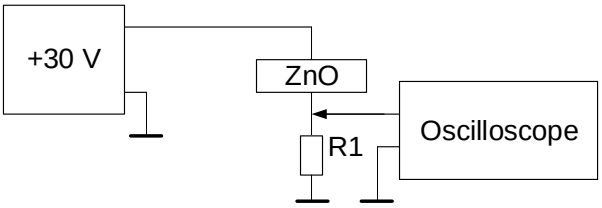

Fig. 2. Scheme for measuring the resistance of the ZnO nanofilm by using a constant current: R1 – measuring resistor 10 MOhm, ZnO – ZnO nanofilm sample.

3. Results and discussion

The paper presents transmission spectra (within the range 300…450 nm) of three samples with deposited ZnO films of different thicknesses. The samples are labeled as Nos 9, 11, and 12. For the samples Nos 9 and 11, spectra were measured on the areas with thicker and thinner films, respectively. In the sample No 12, the region with a thicker film has a too small transmittance coefficient. All the samples differ in the number of layers deposited using the spin-coating technique.

Measured spectra of the samples Nos 9 and 11 from the thicker and thinner sides are presented in Figs 3 and 4.

As can be seen from the graphs, the transmittance coefficient in the areas with a thicker ZnO film is lower by 30 – 40% (in the linear region of the spectrum of pure glass) as compared to the area with a thinner film. Moreover, the drop in the transmittance coefficient within the range 360…390 nm, characteristic of the ZnO spectrum from literature data, is more pronounced for areas with a thinner film, 16.33% as compared to 39.4% for No 9 and 16.86% compared to 36.5% for the sample No 11, respectively. The spectra of samples on areas with a thinner film as compared to pure glass are shown in Fig. 5.

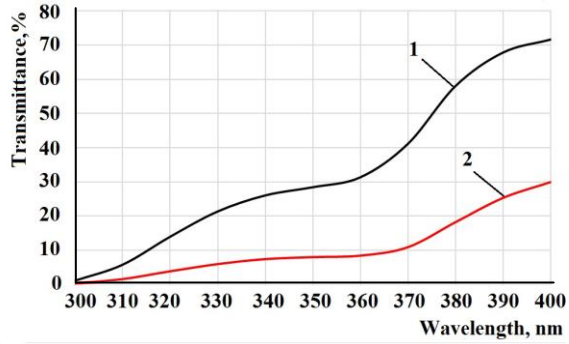

Fig. 3. Transmission spectra of the sample No 9, where *1* corresponds to the side with the film thickness close to 115 nm, and *2* corresponds to the side with the film thickness of about 140 nm.

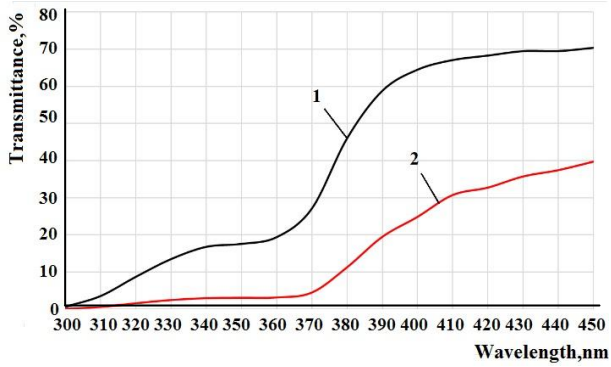

Fig. 4. Transmission spectra of the sample No 11, where *1* represents the side with the film thickness close to 120 nm of ZnO, and *2* represents the side with the film thickness approximately 150 nm.

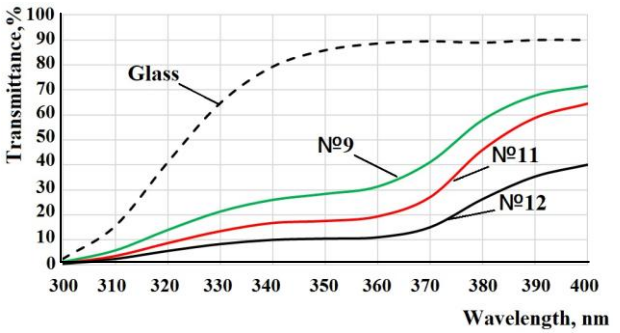

Fig. 5. Transmission spectra of the samples Nos 9, 11 and 12 as compared to the original "Voles" glass.

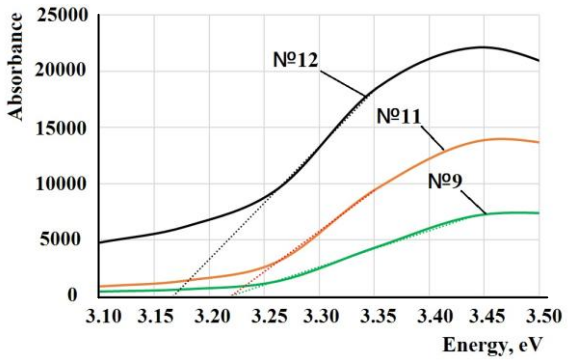

Fig. 6. Absorption spectrum and trend lines for three samples.

The measured spectra revealed a decrease in the transmittance coefficient from Nos 9 to 12, suggesting that the thicknesses of the prepared films differ accordingly.

To calculate the bandgap of the obtained semiconductor film using the Tauc method, the absorption coefficient was recalculated using the formula (1):

$$A(\lambda)=\frac{1}{d}\ln\left(\frac{1}{T}\right),\ \text{cm}^{-1}. \qquad (1)$$

The obtained results of the calculated absorption spectrum and the trend lines for the absorption edge slope are presented in Fig. 6.

As a result of calculations, the bandgap width of the semiconductor film for the sample No 12 is 3.17 eV, and for Nos 9 and 11, it is 3.22 eV, which coincides with the bandgap width of ZnO films known in the literature [7, 26, 27], ranging from 3.15 to 3.35 eV.

Thermo-electric properties of the prepared films were investigated using the oscillographic method with elastic contacts. The stability of the elastic contact was ensured by adhering to the flat-parallel compression method. Otherwise, the substrate surface begins slow movement, and within 1…10 min of measurements, the contact degrades, as evidenced by the oscillogram of the current measurement through the ZnO nanolayer (Fig. 7).

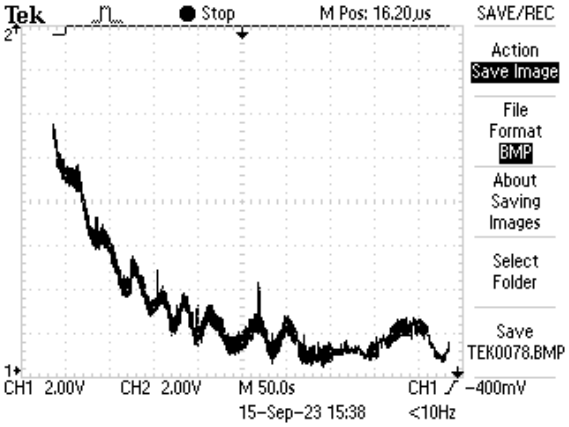

Fig. 7. Oscillogram of the voltage on the measurement resistor demonstrates the degradation process of the elastic contact: current flows from a +30 V source through the nanofilm and a 10 MOhm resistor; the measurement duration is 7 min; at the beginning, the current is 1.1 ± 0.05 μA; after 3 min of observation, the current decreases to 200 ± 50 nA and continues to decrease.

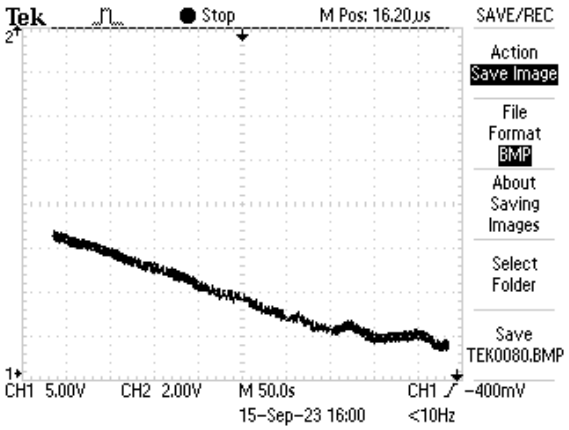

Fig. 8. Linear degradation of the elastic contact due to violation of the flat-parallel compression principle: the current decreased from 1.5 to 0.5 μA in 5 min.

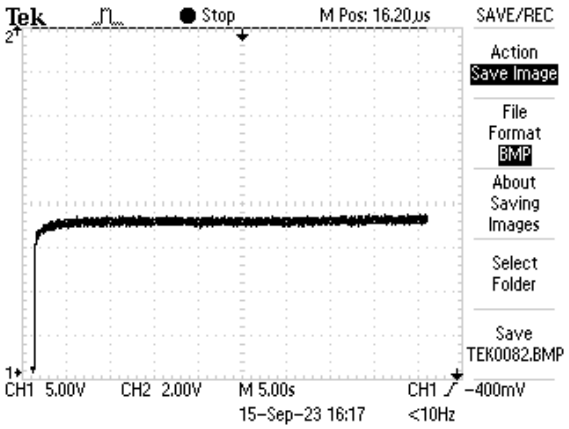

Fig. 9. An example of a stable elastic contact: deployment duration 45 s, current through the ZnO film sample is 1.7 ± 0.05 μA, resistance of the ZnO nanofilm is 7.6 ± 0.2 MOhm, specific resistance is 10.1 ± 0.3 MOhm/□.

Fig. 8 provides another example of degradation of the elastic contact.

Fig. 9 illustrates a stable signal under stable contact conditions.

Fig. 10 depicts the scheme of pulse resistance measurement for the ZnO nanofilm, where on the measurement resistor *R*1, the value of the pulse current is obtained, and then, according to Ohm's law, the resistance of ZnO is calculated.

The energy balance of the nanofilm during the flow of the measuring current and the generation of Joule heat can be estimated under certain preliminary conditions:

- Assuming the average thickness for the investigated samples is 100 nm in the estimation calculation.
- Considering radiative heat transfer (according to the Stefan–Boltzmann law) as the main mechanism of heat transfer to the surrounding space from the film since the thermal resistance of the substrate glass is an order of magnitude higher than the thermal resistance of the film itself, and the conduction mechanism can be neglected.
- Utilizing data for the common material ZnO; for example, taking the specific heat capacitance as 495 J/kg·K (10 times higher than that in glass).

The thermal power *P* of the ZnO nanofilm due to generation of Joule heat is:

$P_1 = IU = 1.7\ \mu\text{A}\cdot(30 - 17)\ \text{V} = 22\ \mu\text{W}.$

Radiation power based on the Stefan–Boltzmann law:

$$P_2 = \sigma\left(T^4 - T_0^4\right)S,$$

where $S = 3\ \text{mm}^2$; $\sigma = 5.67\cdot10^{-8}\ \text{W/m}^2\cdot\text{K}^4$.

The heat exchange balance between the film and surrounding environment: $P_1 = P_2$ or $\sigma S T^4 = P_1 + \sigma T_0^4 S$ (T_0 = 20 °C = 293 K) from:

$$T = \left[\left(P_1 + \sigma T_0^4 S\right)/\sigma S\right]^{1/4} =$$

$$= \left(\frac{22\cdot10^{-6} + 5.67\cdot10^{-8}\cdot293^4\cdot3\cdot10^{-6}}{5.67\cdot10^{-8}\cdot3\cdot10^{-6}}\right)^{1/4} = 294.3\text{K}.$$

Thus, the temperature increase of the ZnO nanofilm (Fig. 9) due to the measuring current flow is 1.3 degrees, which is negligible.

The temperature dependence of the ZnO nanofilm resistance was calculated from the oscillograms of signals obtained according to the scheme in Fig. 10.

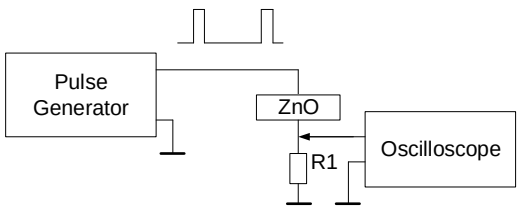

Fig. 10. Scheme for measuring the resistance of ZnO nanofilm in pulse signal mode.

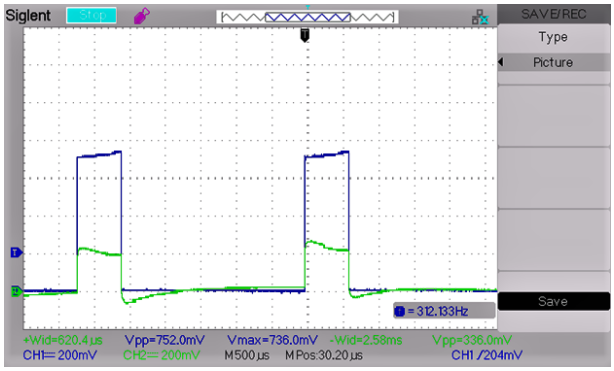

Fig. 11. Oscillograms of signals at the output of generator (channel 1) and on resistor *R*1 (channel 2) in a 1:10 scale: the temperature of ZnO film sample No 4 is 20.2 ± 0.2 °C: generator frequency is 312 Hz, signal duty cycle is 5, the pulse amplitude at the output of generator is 7.6 V, and on resistor *R*1 is 2.2 V.

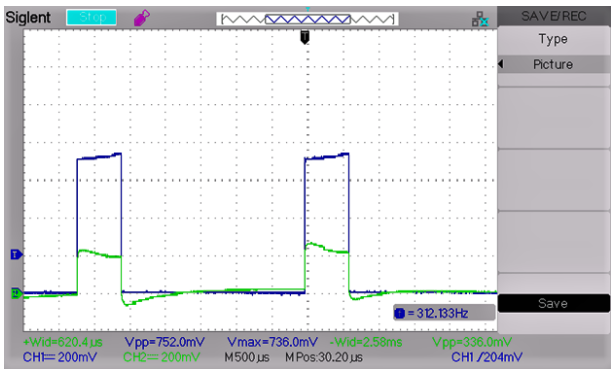

Fig. 12. Oscillograms of signals at the output of generator (channel 1) and on resistor *R*1 (channel 2): the temperature of ZnO film sample No 4 is 58 ± 2 °C; on resistor *R*1, the pulse amplitude increased to 3.6 V.

Figs 11 and 12 show signals at the output of generator (channel 1) and measurement resistor *R*1 for different temperatures. The sample heating was carried out with a 250-W halogen-tungsten lamp, with a heating duration of approximately 40 s.

The resistance of the film at the temperature 20.2 °C is

$$R = \left((7.6\,\text{V} - 2.2\,\text{V})/2.2\,\text{V}\right)\cdot 10\,\text{MOhm} = 24.5\,\text{MOhm}\,.$$

The resistance of the film at the temperature 58 °C is

$$R = \left((7.6\,\text{V} - 3.6\,\text{V})/2.2\,\text{V}\right)\cdot 10\,\text{MOhm} = 18.2\,\text{MOhm}\,.$$

The temperature coefficient of resistance is

$$\text{TCR} = \frac{1}{R_{293}}\frac{\Delta R}{dT} = \frac{1}{R_{293}}\frac{\Delta R}{\Delta T}$$

(for comparison: in copper, TCR is 0.004 K^{-1}, or 42.5 times less).

Overall, when measuring samples with different thickness values of ZnO films, the electrical resistance ranged from 10 to 100 MOhm/□, depending on the film thickness. These measurements allow for estimating the film thickness and its compositional uniformity.

Measurements of the electrical resistance of films, when they are heated from 25 up to 100 °C showed a decrease of approximately 60%.

4. Conclusions

The analysis of transmission spectra of the samples with zinc oxide nanofilms deposited using the sol-gel method allowed determining the energy bandgap in the range of 3.18 to 3.23 eV, indicating the presence of zinc oxide in the nanofilms. The results of these investigations reveal crucial physical properties of the material, confirming its quality and chemical purity.

The surface resistance has also been studied using the elastic contact method. This approach enabled precise measurement of the electrical resistance of high-resistance films with low thermal and mechanical impact on the investigated surface. Our research demonstrated that zinc oxide nanofilms exhibit high resistance, emphasizing their quality and potential for applications in electronics and photoelectronics.

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Оптичні та електричні властивості наноплівок оксиду цинку, нанесених за допомогою золь-гель методу

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Анотація. Дану статтю присвячено дослідженню електричних властивостей наноплівок оксиду цинку (ZnO), отриманих золь-гель методом. Дослідні зразки представляють собою підкладку (25×25×1 мм) із мікроскопного скла марки «Волес» з нанесеними за допомогою золь-гель методу плівок ZnO товщиною в межах 50…150 нм. У ході досліджень було проведено спектральні вимірювання для розрахунку ширини забороненої зони, які підтвердили присутність оксиду цинку у наноплівках та показали їхню оптичну активність. Застосовано осцилографічний метод для вимірювання поверхневого опору наноплівок із застосуванням гальванічного еластичного контакта, що дозволило визначити їхню високу електричну якість та високий опір. Одночасно із цим було встановлено високу механічну міцність наноплівок відносно дії еластичного контакта, які витримували серію із 5…10 вимірювань без помітних змін величини опору. Також було проведено вимірювання зміни електричного опору плівок ZnO при їх нагріванні від 25 до 100 °C. Отримані результати свідчать про придатність цих наноплівок для застосувань у сферах електроніки, фотоелектроніки та сенсорних технологій.

Ключові слова: оксид цинку, золь-гель метод, наноплівка, спектр пропускання, поверхневий опір, еластичний гальванічний контакт.