UDC 621:53.096
DOI: 10.15587/1729-4061.2023.286546

DEVISING TECHNOLOGICAL SOLUTIONS FOR GAS SENSORS BASED ON ZINC OXIDE FOR USE AT CRITICAL INFRASTRUCTURE FACILITIES

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This paper reports a study of a gas sensor based on nanostructured zinc oxide in order to establish the conditions for its production and operating characteristics under the influence of the target gas ethanol. The studied samples were produced by magnetron sputtering on direct current. The method of forming the device structure was chosen among others due to the fact that it has a high rate of deposition at low values of the working gas pressure, there is no overheating of the substrate, a low degree of contamination of the obtained films, the possibility of obtaining samples of uniform thickness on a large area of the substrate. A VUP-5M vacuum unit with an original material-saving magnetron was used to obtain the films. Studies of the effect of temperature on the resistance of a gas sensor based on ZnO have been carried out. It was established that the change in the resistance of the tested sample depends on the temperature of the substrate. The resistance of the gas sensor in atmospheric air decreases with increasing substrate temperature from room temperature (25 °C) to 200 °C. A further increase in temperature from 200 °C leads to an increase in the resistance of the structure until it stabilizes in the temperature range of 300–400 °C. It was established that the operating temperature range of the gas sensor based on ZnO is within 300–400 °C. The characteristics of the gas sensor based on ZnO were studied and the working temperature of the sensor was determined for the rapid identification of ethanol in atmospheric air at a target gas concentration of 500 ppm. It was established that for rapid operation of the instrument structure, the temperature of the substrate should be 400 °C, a decrease or increase in temperature leads to a decrease in the sensitivity of the sensor to the target gas. It was established that the gas sensor demonstrates stability and a consistent sensitivity response upon repeated exposure to the target gas

Keywords: ZnO, gas sensor, magnetron sputtering, operating temperature, sensitivity, reaction stability

Received date 04.06.2023
Accepted date 14.08.2023
Published date 30.08.2023

***How to Cite**: Minska, N., Hvozd, V., Shevchenko, O., Slepuzhnikov, Y., Murasov, R., Khrystych,, V., Strelets, V., Kryvonis, S., Rotar, V., Lypovyi V. (2023). Devising technological solutions for gas sensors based on zinc oxide for use at critical infrastructure facilities. Eastern-European Journal of Enterprise Technologies, 4 (5 (124)), 34–40. doi: https://doi.org/10.15587/1729-4061.2023.286546*

1. Introduction

Under the conditions of a constant increase in the threat of natural, man-made, and military emergencies at critical infrastructure facilities, the problem of controlling toxic, flammable, and explosive gases in the environment has become more acute [1]. The consequences of damage to critical infrastructure objects can extend not only beyond the object itself but also beyond the borders of the country [2, 3]. To control and assess risks in the surveillance zone of potentially dangerous objects, intelligent sensor technology is used, which provides operational environmental monitoring and consists of technical means of information analysis [4]. Technical means of information analysis at critical infrastructure facilities include intelligent sensors, such as dosimeters, gas analyzers, seismic sensors, wireless sensor networks, video recorders, mobile devices, and data received from a satellite [5].

Therefore, devices for monitoring the gas environment in real time are extremely important for timely prevention of emergency situations, protection of the population and the environment [6, 7]. Devices for monitoring the environment, including gas analyzers, must be provided not only to the critical infrastructure object itself but also to the personnel who are at the object and serve it. In the case when the gas analyzer is part of the object's intelligent sensor technology, the dimensions of the gas analyzer are not important in comparison with its sensitivity to the analyzed gas. When it comes to portable gas analyzers for personnel, the following parameters are decisive: the sensitivity of the gas analyzer, its size and weight, and the cost of the gas analyzer. The most common gas analyzers are Honeywell BW Gas Alert-Micro Clip XL (USA), which allows continuous monitoring of up to 4 dangerous gases, and Testo 340 – 4-channel gas analyzer of O_2, CO, NO, NO_2, SO_2 (Germany). Such gas analyzers are also used for setting up any type of fuel combustion equipment that runs on different types of fuel (including coal, fuel oil, or diesel fuel). For carrying out environmental measurements with the calculation of mass and volume emissions. To control the technological parameters of production processes (for example, in the petrochemical and steel industries, in the production of glass, ceramics, building materials, plastics, etc.). Another multi-component gas analyzer that makes it possible to determine dangerous concentrations (combustible gases, carbon monoxide, O_2, H_2S) is the WINTACT WT8811 (manufactured in China). All these gas analyzers have an electrochemical principle of operation. Electrochemical sensors react to gas by changing their electrochemical properties as a result of redox reactions involving the analyzed gas. Such properties include the magnitude of the electric current or the potential of the electrodes. These gas analyzers meet the requirements for the main characteristics of a gas sensor but have a high cost, which limits their mass use as portable gas sensors. For use in gas-dynamic devices for various purposes, including for the creation of portable gas sensors, sensors with a resistive principle of operation are promising. The principle of operation of resistive sensors is based on a change in electrical resistance in the presence of the analyzed gas. Sensors of the resistive type include sensors based on metal resistors and adsorption-semiconductor sensors. Adsorption-semiconductor sensors have advantages among others, namely, the method of their production is suitable for large-scale production, they have a small weight and a large receiving surface. The gas-sensitive layer of such sensors is made of semiconductor oxides. Of special interest are semiconductor oxides SnO_2, ZnO, In_2O_3, Ga_2O_3, TiO_2, WO_3, Fe_2O_3, CeO_2, and perovskites ($BaSnO_3$, $LaFeO_3$, $SrTiO_3$) with different morphologies [8].

The creation of gas sensors based on ZnO is attracting more and more attention due to the variety of methods of obtaining sensitive layers and low cost, where real-time detection of explosive, flammable and toxic gases is required. But despite various studies of ZnO-based gas sensors obtained by various methods, there is no model that would describe the relationship between the operating characteristics and the conditions for creating the sensitive layer. Therefore, it is necessary to carry out further research to establish the specified regularities.

2. Literature review and problem statement

Zinc oxide is a direct-band semiconductor with a large band gap (3.37 eV) and a large exciton binding energy (60 meV), which is a significant advantage among other metal oxides. Zinc oxide is one of the first to be used in electronics, optoelectronics, and laser technology, which is due to the set of its unique physical and chemical properties. Such properties include chemical and mechanical stability, availability, low cost, photosensitivity, and high radiation resistance [9, 10]. Since ZnO is a chemiresistive sensor, the change in its resistance depends on the presence of chemisorbed oxygen ions. In the presence of atmospheric air, oxygen molecules are adsorbed on the surface of ZnO and on the boundaries of crystal grains, creating potential barriers [11, 12]. In particular, oxygen molecules adsorbed on the ZnO surface capture free electrons and turn into ions $\left(O_2^{2-}\right)$, which form a depleted layer near the ZnO/air interface, and as a result, the conduction band and valence band are bent. The formation of the depleted layer has a decisive effect on the electrical resistance of the semiconductor. Due to the extraction of electrons from the conduction zone of ZnO, the resistance of ZnO increases. If the reducing gases interact with the chemisorbed oxygen ions on the ZnO surface, the resistance decreases because the oxygen ions donate free electrons to the ZnO conduction band.

Given that in the presence of atmospheric air, oxygen molecules are adsorbed both on the surface of ZnO and on the boundaries of crystal grains, the morphology of the ZnO layer affects the change in the electrical resistance of the structure. Accordingly, the morphology of the ZnO layer affects the parameters of the gas sensor based on it.

The structure of ZnO can take on various morphological forms. Among others, one-dimensional structures are known – needles, spirals, nanorods, wires, ribbons, and combs. ZnO can exist in the form of two-dimensional structures such as nanoplates and nanobeads. In addition, ZnO can be used in the production of a wide range of three-dimensional structures, such as flowers, snowflakes and other shapes. Paper [13] describes the production of zinc oxide nanoparticles using surfactants in the form of various precursors and modification of the ZnO surface using polyvinyl alcohol (PVA). The authors of the work established a regularity between the morphology of the obtained ZnO samples and the method of forming the sensitive layer. However, the question of the dependence of the working characteristics of the gas sensor on the morphology of the sensitive layer has not been resolved. The authors of paper [14] obtained ZnO samples with a porous mesh structure with oxygen vacancies using the electrospinning method. The resulting structures demonstrated high sensitivity and reaction speed, as well as selectivity to acetone vapors. The work does not indicate the stability of the gas sensor during repeated exposure to the target gas, which is an important operating characteristic of the instrument structure. In [15], the authors studied MoS_2 nanosheets covered with ZnO nanoparticles, which were produced by a two-stage hydrothermal method. The authors found that the sensor based on MoS_2 nanoplates covered with ZnO nanoparticles exhibits high sensitivity to ethanol, and it may have potential applications for the detection of ethanol vapors, thanks to the support substrate of specific two-dimensional MoS_2 nanosheets. But questions related to such characteristics as the stability of the gas sensor, reaction speed and relaxation speed remained unresolved. The authors of [16] synthesized Ag-doped ZnO nanoparticles grown in high density with an average diameter of ~20±5 nm using a light hydrothermal process. Synthesized Ag-doped ZnO nanoparticles were used as a functional material to fabricate an effective ethanol gas sensor, which demonstrated high sensor performance. The optimal working temperature of the gas sensor was established at 320 °C and a plausible gas detection mechanism was presented. The research was conducted at a gas concentration of 200 ppm. The behavior of the sensor at higher concentrations of the target gas was not investigated in the work, which creates limitations for the use of the obtained results. Paper [17] demonstrated a highly efficient ozone gas sensor based on hollow In_2O_3 microtubes obtained from metal-organic frameworks (MOFs) decorated with ZnO nanoparticles. The operating temperature of the sensor is determined at 150 °C. It was established that the gas detection limit of the In_2O_3/ZnO sensor is only 25 ppm. Also, such a sensor exhibits strong gas response, excellent response and recovery time, selectivity, stability, and repeatability. However, the stability of the operating characteristics at a high concentration of the target gas has not been investigated. The described methods of obtaining nanostructures of zinc oxide are expensive, at the same time, the method of direct current magnetron sputtering has a low cost. With the help of the direct current magnetron sputtering method, it is possible to create uniform films on a large surface area, this method is easy to control. When using this method to create nanostructures based on zinc oxide, it is possible to adjust the parameters of device structure formation, such as pressure, temperature of the substrate, duration of deposition, and distance from the target to the substrate. These parameters optimize the sensitivity of the sensor. In work [18], the deposition parameters were as follows: the working pressure of the argon-air mixture during the spraying process is $(2.1–2.6)\times10^{-2}$ mmHg. The substrate temperature was 300 °C. As a result of the research of the obtained samples, it was established that the change in the resistance of the tested sample is proportional to the change in the concentration of the target gas. After the surface of the sensor becomes saturated with adsorbed molecules, the resistance no longer decreases, even if the concentration of the gas continues to increase. The reaction of the gas sensor to the target gas - ethanol at a concentration above 150 ppm was almost absent.

The review of studies aimed at the formation of a sensitive layer for gas sensors based on ZnO allowed us to single out the magnetron sputtering method on a direct current as one of the promising methods for large-scale production. It was established that the working characteristics of the gas sensor are influenced by both the conditions of deposition by this method and the working temperature of the instrument structure. In addition, the performance characteristics of the gas sensor vary depending on the concentration of the target gas.

The lack of physical models of optimal ZnO-based gas sensors and comprehensive work on their experimental testing predetermines the need for research aimed at determining the conditions of its production and operating characteristics under the influence of the target gas.

3. The aim and objectives of the study

The purpose of this work is to devise technological solutions for the formation of a ZnO-based gas sensor to increase the sensitivity and stability of operation during repeated exposure to the target gas. This will make it possible to create semiconductor gas sensors of the explosive ethanol gas using simple and suitable for large-scale production methods.

To achieve the goal, the following tasks must be solved:

– to investigate the effect of temperature on the resistance of a gas sensor based on ZnO obtained by the method of direct current magnetron sputtering and to determine its operating temperature;

– to investigate the characteristics of a gas sensor based on ZnO obtained by magnetron sputtering on a direct current at operating temperature.

4. The study materials and methods

4. 1. The object and hypothesis of the study

The object of our research is a gas sensor based on nanostructured zinc oxide.

Depending on the deposition conditions of the sensitive layer of the gas sensor based on zinc oxide, the surface of the ZnO nanostructures ends with zinc or oxygen ions. This determines the conditions of its use at different temperatures. The resistance of nanostructures based on oxide semiconductors decreases as the operating temperature increases to a certain value, which is associated with intensive adsorption of oxygen on its surface. When the equilibrium between the thermal excitation of electrons and the adsorption of oxygen is reached, the change in temperature does not significantly affect the change in resistance until the established equilibrium is disturbed. The range of operating temperatures at which the resistance of the gas sensor does not change significantly will be considered operating for the analysis of sensitivity to the target gas.

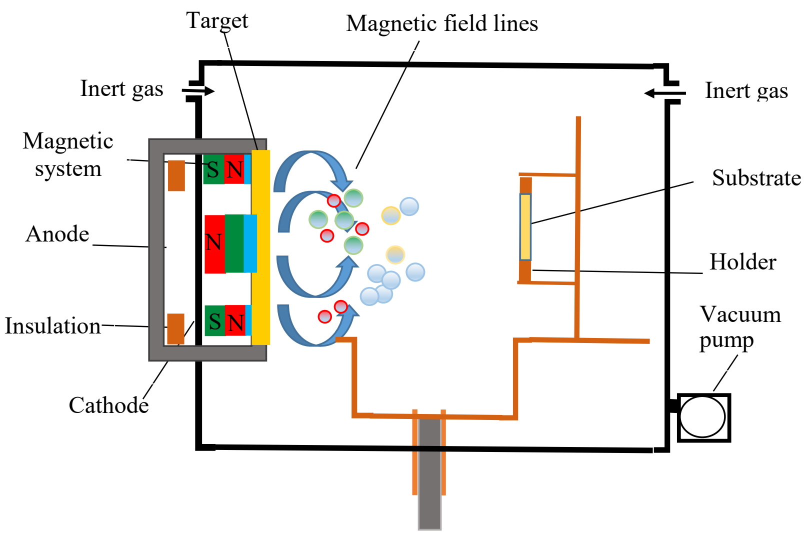
4. 2. Obtaining nanostructured zinc oxide by magnetron sputtering at direct current

Obtaining the tested samples of the gas sensor based on ZnO was carried out by the method of direct current magnetron sputtering. To obtain the sensitive layer of the gas sensor, a VUP-5M vacuum unit was used (Fig. 1).

The target for obtaining ZnO on the surface of the substrate was a zinc target (99.99 % pure).

The length of the discharge gap, which is the distance between the magnetron and the substrate, was 70 mm. The power of the magnetron was 0.2 W/cm^2. High-purity argon was used as an inert gas, and oxygen as an active gas. The outlet pressure in the vacuum chamber was 3×10^{-5} mmHg, the working pressure of the argon-air mixture during the spraying process was $(2.1–2.6)\times10^{-2}$ mmHg. The substrate temperature was 350 °C. The deposition rate was 12 Å/s. [19] A helium-neon laser with a wavelength of 633 nm was used to determine the thickness of the films. The resulting films had a thickness of 2 μm.

a



b

Fig. 1. Obtaining the studied samples of the gas sensor based on ZnO: *a* – general view of vacuum installation VUP-5M; *b* – a schematic representation of the process of obtaining the tested samples of the gas sensor

4. 3. Investigation of the main characteristics of a gas sensor based on ZnO

The main characteristics of the functioning of the gas sensor include sensitivity, response time, relaxation time, stability, and operating temperature.

The study of the main characteristics of the gas sensor based on ZnO was carried out using the installation, the block diagram of which is shown in Fig. 2 [18].

The measuring device consisted of a source of constant voltage, a digital multimeter, and a controller. The studied sample of the ZnO-based gas sensor was placed in a glass box with a volume of 3.6×10^{-2} m^3. The source of constant voltage provided the voltage between the two electrodes at the level of 5 V. As a heating element for the studied sample, a metal layer of Pt/Ti deposited on the substrate by magnetron sputtering was used.

The indicator of the current flowing through the gas sensor was measured using a multimeter. Ethanol (C_2H_6O) was used as the target gas. The choice of the target gas is due to the expansion of the spectrum of its application in various industries, in chemical, light, food, automotive. Analysis of class B fires, which include alcohols, showed that they have a tendency to increase both in number and scale, ethanol is a fire hazard gas.

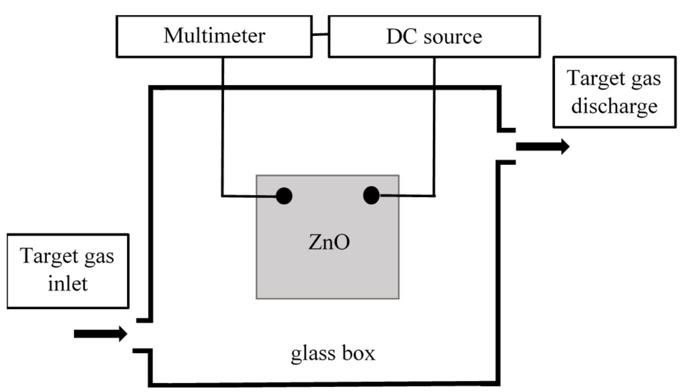


Fig. 2. Block diagram of the installation for studying the sensitivity of the gas sensor

The glass box was filled with the studied gas using an air pump. The target gas concentration was controlled by flow rate and delivery time at ambient temperature. Relative humidity was maintained at 50 %. Target gas concentration, 500 ppm.

The resistance of the gas sensor based on ZnO obtained by the method of direct current magnetron sputtering was determined at temperatures from 25 °C to 450 °C. The temperature of the heating element was varied and controlled by changing the applied voltage using a DC source, and the temperature was measured using a thermocouple attached to the surface of the device next to the sensitive area. The stability of the measured temperatures was about ±2 °C. To obtain experimental values, 12 parallel experiments were conducted.

To determine the sensitivity of the gas sensor, an air-water mixture was fed into the chamber of the measuring unit until a stable value of the sensor signal measured with a multimeter was established. After that, the target gas was supplied to the chamber until the specified concentration was established. After establishing a constant value of the signal, the value of the multimeter was fixed. Measurements were repeated for each of the selected concentrations. The value of sensitivity (S) was calculated as:

$$S = \frac{R_à}{R_g - R} 100\,\%, \qquad (1)$$

where R_a is the resistance of the gas sensor in air, R_g is the resistance of the gas sensor in the atmosphere of the target gas.

To establish the speed of the gas sensor, an air-water mixture is fed into the chamber of the measuring unit until a stable value of the sensor signal is measured with a multimeter. After that, the target gas is fed into the chamber every 10 seconds. the value of the multimeter indicator was fixed until a constant value of the signal was established.

5. Results of the study of the main characteristics of the gas sensor based on ZnO

5. 1. Results of the study of the influence of temperature on the resistance of a gas sensor based on ZnO

The results of the research into the resistance of the gas sensor based on ZnO are given in Table 1. Resistance measurements of the ZnO-based gas sensor in the range from room temperature to 450 °C were measured in humid air at a constant relative humidity of 50 %.

Table 1

Change in resistance of ZnO-based gas sensor depending on changes in substrate temperature

No. of entry	Temperature (t), °C	Resistance (R_a), MOhm	Humidity (W), %
1	25	30	50
2	50	28	50
3	100	2.8	50
4	150	0.5	50
5	200	0.2	50
6	250	0.5	50
7	300	2	50
8	350	3	50
9	400	2.5	50
10	450	2	50

The temperature dependence of the resistance of the gas sensor based on ZnO is shown in Fig. 3.

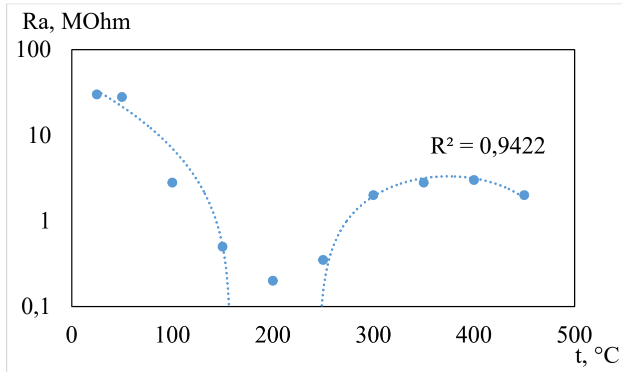


Fig. 3. Dependence of ZnO-based gas sensor resistance on substrate temperature

As can be seen from Fig. 3, when the temperature rises to 200 °C, there is a sharp decrease in the resistance of the sample, a further increase in temperature leads to an increase in resistance until it stabilizes in the temperature range of 300–400 °C.

5. 2. Investigation of the characteristics of a gas sensor based on ZnO at operating temperature

The sensitivity of the gas sensor was determined by measuring the resistance depending on the temperature in the range of 200–450 °C and the ethanol concentration of 500 ppm, followed by calculation according to formula (1). The change in sensitivity depending on the operating temperature is shown in Fig. 4.

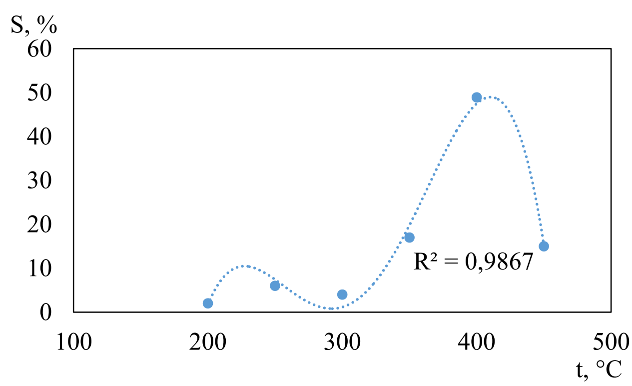


Fig. 4. ZnO-based gas sensor sensitivity

As can be seen from Fig. 4. the sensitivity of the ZnO-based gas sensor changes when the operating temperature changes. The maximum sensitivity is observed at a substrate temperature of 400 °C.

To analyze the stability of the response of the sensitivity of the ZnO-based gas sensor to the target ethanol gas, the dependences shown in Fig. 5 were constructed.

The repeatability of the sensitivity response was studied by measuring the resistance of the ZnO-based gas sensor in an atmosphere of a target gas with a concentration of 500 ppm at an operating temperature of 400 °C.

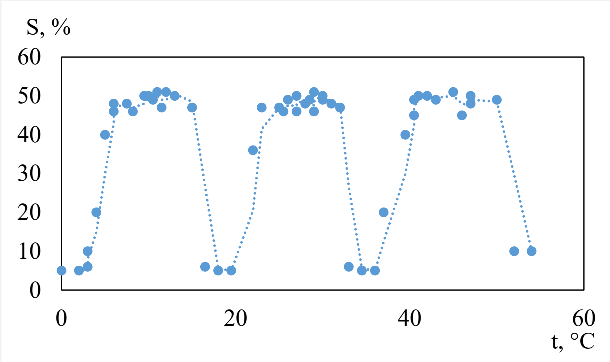


Fig. 5. Response of the sensitivity of the ZnO-based gas sensor to the target gas under repeated exposure (500 ppm gas concentration)

6. Discussion of results of investigating the gas sensor based on ZnO obtained by magnetron sputtering on direct current

In the work, a gas sensor based on ZnO obtained by the method of direct current magnetron sputtering was investigated. This method of forming the instrument structure was chosen among others due to the following advantages. The DC magnetron sputtering method has a high deposition rate at low working gas pressure values. In this method, there is no overheating of the substrate, a low degree of contamination of the obtained films, the possibility of obtaining samples of uniform thickness on a large area of the substrate. In the work, the effect of temperature on the resistance of a ZnO-based gas sensor was studied. As can be seen from Fig. 3, the resistance of the gas sensor in ambient air decreases with increasing temperature of the substrate from room temperature (25 °C) to 200 °C. A further increase in temperature from 200 °C leads to an increase in the resistance of the structure until the moment of its stabilization in the temperature range of 300–400 °C. The increase in the resistance of the sample at a temperature above 200 °C is associated with the intensification of oxygen adsorption on the surface of the film, the stabilization of the sample resistance in the temperature range above 300 °C is due to the establishment of an equilibrium state. Thus, it was established that the operating temperature range of the gas sensor based on ZnO obtained by the method of direct current magnetron sputtering is within 300–400 °C. To further narrow the operating temperature range, we obtained the sensitivity values of the sample in the atmosphere of the target gas - ethanol. As can be seen from Fig. 4, the maximum sensitivity of the ZnO-based gas sensor to the target gas of ethanol is observed at a substrate temperature of 400 °C, further increasing the temperature leads to a decrease in the sensitivity of the sample. Thus, it was established that the operating temperature of the gas sensor under study reaches 400 °C. The obtained results of the study of the ZnO-based gas sensor on the repeatability and stability of operation demonstrated that the sensitivity response of the ZnO-based gas sensor to the target gas is repeated. It was established that the ZnO-based gas sensor works stably (Fig. 5).

Unlike [18], where the response of the sensor to the target gas at a concentration above 150 ppm was almost absent, the result obtained in our study showed a high sensitivity of the sensor at a concentration of 500 ppm (Fig. 4). This was made possible by setting the optimal operating temperature of the gas sensor at 400 °C.

The operating characteristics of the ZnO-based gas sensor were determined under the condition that the sensor was exposed to only one target gas. The values of the operating characteristics of the sensor when it is exposed to several gases at the same time are unknown. Therefore, the condition of applying the research results is the use of only one target gas.

The lack of results of the reaction speed and recovery speed of the gas sensor upon repeated exposure to the target gas can be attributed to the shortcomings of this study. This shortcoming is planned to be eliminated during the next research. Further studies of the ZnO-based gas sensor obtained by direct current magnetron sputtering will also ffocus on studying the performance characteristics of the gas sensor in the atmosphere of a mixture of volatile organic compounds. It is necessary to establish the regularities of the reaction of the gas sensor to a certain gas in the composition of the mixture.

7. Conclusions

1. Samples of a gas sensor based on ZnO were obtained by the method of direct current magnetron sputtering. It was established that the change in the resistance of the tested sample depends on the temperature of the substrate. It was established that the operating temperature range of the gas sensor based on ZnO is within 300–400 °C.

2. The characteristics of the gas sensor based on ZnO, obtained by the method of direct current magnetron sputtering, were studied and the working temperature of the sensor was determined for the rapid identification of ethanol in atmospheric air. It was established that for rapid operation of the instrument structure, the temperature of the substrate should be 400 °C, a decrease or increase in temperature leads to a decrease in the sensitivity of the sensor to the target gas. It was established that the gas sensor works stably with a consistent response of sensitivity under repeated exposure to the target gas at the level of 50 %.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

Funding

The study was conducted without financial support.

Data availability

The data will be provided upon reasonable request.

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