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DETERMINATION OF INDICATORS OF METHANE DETECTING THERMOCATALYTIC SENSOR

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Abstract: As a result of research conducted in the work, a highly sensitive, selective chemical sensor for detecting natural gas was developed using a gas-sensitive film with a semiconductor layer composition consisting of zinc and cobalt oxide. Timely detection of natural gas as a result of using the sensor; to prevent the risk of fire; to reduce dangerous situations in production; to build an automated gas safety system; creation of a microclimate system is achieved.

Keywords: natural gas, methane, metal oxide semiconductor sensor, zinc oxide, cobalt oxide, gas-sensitive material resistance, signal concentration dependence.

The interest in the natural gas (methane) sensor is undoubtedly due to its wide application in ecology and industry in ensuring the safety of equipment. Currently, metal oxides are widely used as sensitive elements of semiconductor sensors (SSIs). As a result of the research, it was recommended to use a gas-sensitive film consisting of zinc and cobalt oxide as a gas-sensitive semiconductor layer for detecting natural gas [1]. For this reason, the developed natural gas semiconductor sensor consists of semiconductor gas sensitive materials (GSMs) based on zinc and cobalt oxides coated with spirals made of platinum microwire [2]. The principle of operation of the sensor is based on the fact that the electrophysical properties of its gas-sensitive layer change in accordance with the content of the analyzed gas. As a result of the conducted research, a selective and sensitive semiconductor sensor was developed to determine the concentration of natural gas from the composition of atmospheric air and gas mixtures [2].

The purpose of this work, is to determine the sensitivity, selectivity, expressivity of the sensor and the dependence of its signal on concentration and temperature.

In the course of research, the metrological characteristics of natural gas sensors with sensitive elements made on the basis of zinc and cobalt oxides were thoroughly studied.

The effect of temperature on the sensitivity of a semiconductor natural gas sensor over a wide range was investigated. It is known that the rate of adsorption, desorption and reaction of natural gas on the semiconductor surface depends on the temperature. The temperature of the gas-sensitive layer of the natural gas sensor is ensured by changing the value of the voltage applied to it [3].

The optimal temperature value for heating the gas-sensitive material is determined by the maximum sensitivity of the sensor to the gas. The study of temperature dependence of YAOS-SN4 sensitivity to gas was carried out dynamically in the temperature range from 200 0C to 500 0S with a difference of 50 0S. The experiments were carried out in the following sequence:

1. Setting the desired temperature in the chamber for determining the effect of temperature on the sensitivity of the sensor and waiting for the temperature to stabilize.

2. After the temperature stabilizes, send a flow of fresh air using a suitable valve and measure the GSM resistance.

3. Introduction of natural gas into the chamber. To do this, the air valve is closed and the analyzed gas mixture valve is opened. The steady state of the signal value corresponding to the given concentration value of the component is determined.

4. The gas supply valve is closed and the air valve is opened. The time to return the resistance of the sensor to the initial value of ±10% is determined.

At this temperature, the signal value corresponding to the same SN4 concentration of GSMs based on SiO2/ZnO increases with increasing CoO content in GSM from 1-10% and reaches its maximum value in GSM with SiO2/(90%ZnO+10%CoO). At the optimum temperature (350 0C), the highest signal for SN4 was observed in GCM based on SiO2/(90%ZnO+10%CoO).

As the temperature deviates from the optimal value, a decrease in the useful analytical signal was observed. If the temperature is large, it reduces the sensitivity due to the reduction of oxygen and gas adsorption. It should be noted that high temperatures can cause the sensor to break down the GSM and cause it to fail[4].

The results of studying the sensor temperature dependence of GSM resistance prepared on the basis of SiO2/(90%ZnO+10%CoO)+PEG nanocomposite are shown in Fig. 1b. As a result of the conducted research, when using SiO2/(90%ZnO+10%CoO) semiconductor films obtained with PEG, we estimate the optimal temperature value of -2800C, which provides the maximum signal of GSM compared to natural gas. Thus, the use of GSM containing SiO2/(90%ZnO+10%CoO) +PEG causes the temperature value of the maximum signal of the natural gas detection sensor to decrease from 3500C to 2800C, i.e. by 700C.

For this reason, further experiments involving sensors prepared using GSM with SiO2/(90%ZnO+10%CoO)+PEG and SiO2/(90%ZnO+10%CoO) at values of 2.1 V and 2.8 o was carried out. The change in the resistance of NAO according to different values of temperature is explained by the different adsorption of gases on the GSM surface and the different mechanisms of their interaction, and this can be used for selective detection of SN4 in the presence of other gases.

The time required for the sensor signal to reach its maximum value and return to the initial value in natural gas detection characterizes the sensitivity of the sensor and is determined experimentally. The dynamic indicator of the sensor is the time required to reach 90% of its signal value for a given concentration. This time is determined in the form of totk or t09, its return to the initial value (recovery) time is the remaining value of 10% of its signal in the air after the gas supplied to the sensor is stopped. Sensor recovery time is indicated by ttik or t01. During the experiments, the dynamic performance of sensors developed on the basis of gas-sensitive films SiO2/(90%ZnO+10%CoO) SiO2/(90%ZnO+10%CoO)+PEG at a temperature of 350 0C was thoroughly studied. The results of this study are presented in Table 5.1.

SiO2/(90ZnO+10%CoO) and SiO2/(90ZnO+10%CoO)+PEG based sensors have a minimum time of 18-23 seconds to reach a high signal value. The stabilization time (t0.9) of the sensors is 15 seconds for SiO2/(90ZnO+10%CoO) and 11 seconds for the sensor based on SiO2/(90ZnO+10%CoO)+PEG. This value (value of t09) for a gas-sensitive layer based on zinc oxide (SiO2-ZnO) without a catalytic layer was 28-30 seconds, and the recovery time of the sensor readings was more than 1 minute. It should be noted that the results observed in the study correspond to the theoretical ideas about the mechanism of change in the resistance of the gas-sensitive layer, that is, a decrease in resistance is observed when interacting with gas, and the resistance is restored after the gas exposure is stopped. the extended experiments show that GSM made on the basis of zinc and cobalt oxides with the presence of PEG can be used for express detection of natural gas, which allows the sensor developed on the basis of GSM to be used for express control of fire-hazardous situations in closed ecological systems.

The sensitivity of the semiconductor GSM is expressed by the change in the resistance (R) or electrical conductivity (s) of the material when it is exposed to a certain concentration of analyte gas. A change in resistance (conductivity) occurs as a result of a number of sequential physicochemical processes occurring on the surface. Continued adsorption processes on the surface of GSM are associated with changes in the electronic state of the surface, which leads to surface conductivity. When zinc oxide is added to the silicon oxide film resulting from the hydrolysis of tetroethoxysilane (TEOS), its sensitivity to natural gas increases. Later, sensors sensitive to natural gas were obtained by adding zinc and cobalt oxide to the composition of GSM [1,5.]. Usually one of the oxides (ZnO) is the main one by mass. The latter, i.e., the addition of (CoO) in a relatively small amount improves the gas-sensing and selectivity properties of the oxide, and generally improves the performance of the sensor. To obtain a complex gas-sensitive material, the dopant was added at the sol-gel transition stage of the solution. The dopant was added to the solution in the form of cobalt chloride. From the results of studying the sensitivity of SiO2/ZnO-CoO-based films with 1-15% CoO in the detection of natural gas, high sensitivity to the detected component was observed in the SiO2/(90%ZnO+10%CoO) gas sensitive material. The addition of 1 % CoO to the SiO2/ZnO-based GSM resulted in a 1.5-fold increase in the natural gas sensitivity of the sensor. Increasing the amount of SoO in GSM to 5 and 10% increased the sensitivity of the sensor to natural gas by 3.6 and 5.7 times, respectively.

In the next experiments, the comparative characteristics of the sensitivity of the sensors developed on the basis of the synthesized SiO2/(90%ZnO+10%CoO) films with and without polyethyleneglycol were studied. The results of testing the sensitivity of films based on SiO2/(90%ZnO+10%CoO) and SiO2/(90%ZnO+10%CoO) +PEG in the process of natural gas detection are presented in Figure 3. From the results presented in the figure, the linear range of the sensor signal in the process of natural gas detection is SiO2/(90%ZnO+10%CoO) for the sensor made on the basis of the SiO2/(90%ZnO+10%CoO) film starting from 80 mg/m3 SiO2/(90%ZnO+ We see that it corresponds to 30 mg/m3 for the sensor based on the film containing 10%CoO)+PEG. From these studies, it was observed that the sensitivity of the film synthesized in the presence of PEG to natural gas was 2.7 times higher than that of the film obtained without polyethylene glycol. The obtained results show that the use of SiO2/(90%ZnO+10%CoO)+PEG film in the process of natural gas (natural gas) detection significantly increases the sensor sensitivity.

The study of the change of the sensor signal according to different concentrations of natural gas was carried out in a special device. Determining the sensitivity to natural gas at the optimal temperature was carried out by measuring the resistance of the gas-insensitive material in the environment of mixtures of different concentrations of natural gas in air.

It was observed that the resistance of the sensor made of SiO2/(90%ZnO+10%CoO) axis decreased to 280 kOhm (from 3530 to 3250 kOhm) at the starting points of the experiment with natural gas concentration up to 500 ppm. A further increase of natural gas to 1500 ppm causes the resistance to decrease to 260 kOhm (3250 to 2990 kOhm).

This representation of the concentration dependence of the signal causes a number of inconveniences in signalizer production and fundamental research. It is more convenient to express the resistance of the sensor in a normalized form or logarithmically. Using conductance instead of resistance as a measured parameter has several advantages in visualizing the concentration dependence of the signal in the gas analyzer signal processing scheme. In addition, the electrical properties of the sensor depend on the structure of the gas-sensitive layer, its physical and chemical properties, and the mode of its formation. In experiments, the concentration of natural gas is 0.1-3.0 haj. The dependence of the signal of sensors developed on the basis of SiO2/(90%ZnO+10%CoO)+PEG and SiO2/(90%ZnO+10%CoO) in the range of % on the amount of gas mixture was studied. The results of this study are presented in Fig. 4 .

For comparison, a film containing SiO2/ZnO was obtained. Gas sensors with SiO2/ZnO-CoO composition showed the highest sensitivity to natural gas.

The fact that the dependence of the signal (mV) on the concentration has a straight linear character allowed the development of a device with a simple construction for measuring the amount of natural gas. Thus, in experiments conducted in a wide range of natural gas concentrations, the dependence of the signal of the sensors developed on the basis of films containing SiO2/(90%ZnO+10%CoO)+PEG and SiO2/(90%ZnO+10% CoO) on the amount of natural gas was studied. In this case, the sensor developed on the basis of a film consisting of SiO2/(90%ZnO+10%CoO)+PEG showed higher sensitivity than the gas sensor based on the use of a nanocomposite film made of SiO2/(90%ZnO+10%CoO) without polyethylene glycol. Under the same conditions, the sensitivity of the sensor obtained in the presence of polyethylene glycol was found to be 1.92 times higher than the sensitivity of the sensor prepared on the basis of the film obtained without polyethylene glycol.

Experiments showed that the results obtained at a temperature equal to 350 0C range from 0.1 to 3.0%, and the dependence of the signal on the concentration of natural gas in the mixture has a straight line.

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