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Technological process of manufacturing a gas-sensitive multisensor chip based on a passivating coating of zinc oxide nanorods obtained by thin-film technology

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Abstract. A method for manufacturing a gas-analytical multisensor chip is presented. The technological process of manufacturing with an additional SiO_2 layer is described. It was found that the design of the experimental sample of the multisensor chip and the technology of synthesis of low-dimensional gas-sensitive layers provide sensitivity to detected gases up to 1 ppm and allow to increase the speed and temporal stability.

Keywords: gas-sensitive layer, dopant, response time, recovery time, technological process

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Технологический процесс изготовления газочувствительного мультисенсорного чипа на основе пассивирующего покрытия наностержней оксида цинка, полученных по тонкопленочной технологии

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Аннотация. Представлен способ изготовления газоаналитического мультисенсорного чипа. Описан технологический процесс изготовления с дополнительным слоем SiO_2. Установлено, что конструкция экспериментального образца мультисенсорного чипа и технология синтеза низкоразмерных газочувствительных слоев обеспечивают чувствительность к детектируемым газам до 1 ppm и позволяют повысить быстродействие и временную стабильность.

Ключевые слова: газочувствительный слой, легирующая добавка, время отклика, время восстановления, технологический процесс

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Introduction

Despite their high gas sensitivity and long-term stability, existing methods for manufacturing a gas multisensor based on zinc oxide have obvious drawbacks. In one method, a zinc oxide layer is obtained by electrochemical deposition on a dielectric substrate with strip electrodes, to which a constant electric potential is applied. The disadvantage is that it is impossible to completely control the filling of the zinc oxide structures of the gaps between the strip electrodes, which demonstrates the lag of the electrochemical deposition method compared to the hydrothermal method of zinc oxide deposition. In another method, the heaters are placed on the opposite side from the gas-sensitive layer, which leads to a longer heating due to the heat flow to the heater through the substrate [1].

Thus, there is a problem of creating a gas-analytical multisensor chip, the chemoresistive elements of which are made on the basis of zinc oxide nanostructures, the synthesis of which is carried out by the hydrothermal method with high temporal stability and increased speed. Such a multisensor chip should be distinguished by an increased service life at a low cost and is capable of detecting organic vapors with different concentrations.

Experimental technique

During the implementation of the technology for obtaining a gas-sensitive multisensor based on zinc oxide nanorods, sitall St 50–1–1, which has low thermal conductivity, was taken as the material of the dielectric substrate [2]. At the first stage, the substrate is cleaned and the surface is prepared. At the second stage, a set of meander heaters is formed from Pt using the thermal vacuum deposition method. At the third stage, a dielectric heat-transfer layer is deposited from polycrystalline SiC with a thickness of 1.5–2 μm, which has low oxide formation energy with high thermal conductivity. This allows increasing the heating rate of the gas -analytical layer. At the fourth stage, a set of coplanar interdigital electrodes is formed. At the fifth stage, zinc oxide nanorods obtained by hydrothermal synthesis are deposited. The technological process is shown in Figure 1.

The nanorods are synthesized as follows. The first seed layer of zinc oxide is applied by magnetron sputtering methods with a thickness of about 20–50 nm. To form the embryonic layer, a solution was prepared by mixing 50 ml of isopropyl alcohol and 0.05 g of dihydrate zinc acetate. This solution was first applied in a single layer (10 μl), then subjected to centrifugation at 3000 rpm for 60 seconds, followed by annealing at 350 °C for 2 minutes. This layer application process was repeated three times. Accordingly, the embryonic layer consists of a base of three layers of 10 μl each, applied using the described technology. To grow the second structured functional layer of ZnO nanostructures, zinc acetate (175.6 mg), water (80 ml), hexamethylenetetramine (112.1 mg) and centrimonium bromide (27.6 mg) are mixed. Substrates with the seed layer are dipped into the resulting solution. Synthesis is carried out in a thermostat at a temperature of 85 °C for 1 hour. Then the substrates are washed with distilled water and dried at room temperature. Then, annealing is performed and ZnO nanorods with a SiO_2 film, followed by etching to the level of colloidal quantum dots are passivated. The SiO_2 film, which provides protection from oxidation, is obtained by RF magnetron sputtering [3].

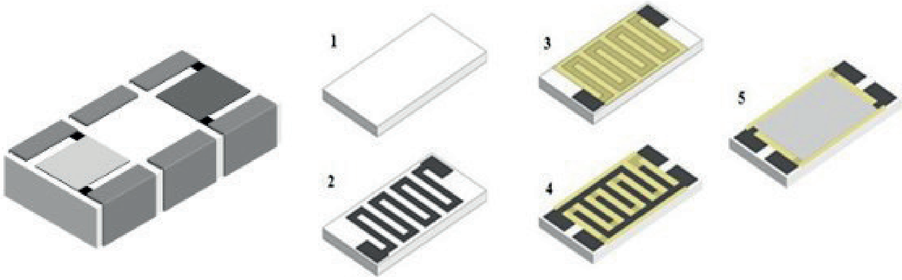

Fig. 1. Scheme of the sensor layer structure synthesis

Results and Discussion

The analysis of the obtained gas-sensitive multisensor chip was conducted using scanning microscopy on an atomic force microscope (AFM) platform NTEGRA, which allows contact and semi-contact modes. However, it was more appropriate to measure the samples using the more complex semi-contact mode, due to the inevitable damage to fragile nanorods in contact mode.

Analysis showed that the growth pattern of nanorods on the metal and on the embryonic layer differs [4]. Nanorods grow not only on the embryonic layer (the substrate itself) but also throughout the volume of the solution. However, nanorods growing within the solution volume eventually fall onto the surface of the substrate [5]. Such rods do not participate in electrical conductivity (they are not anchored) and, therefore, do not affect gas sensitivity.

Nanorods were studied under the influence of isopropanol vapors of concentrations equal to 1150 ppm and 5370 ppm (Fig. 2).

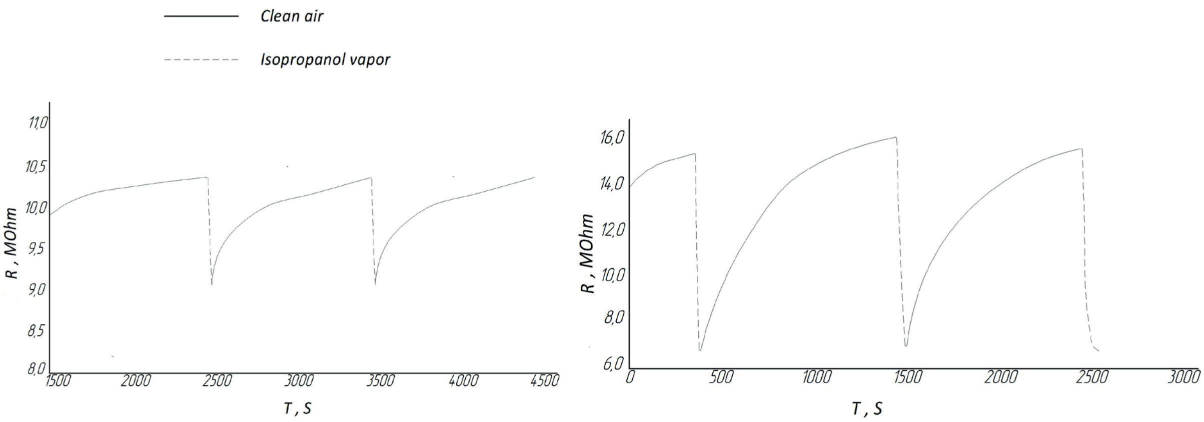

Fig. 2. A graph of resistance dependence upon exposure to isopropanol vapors of concentrations equal to 1150 ppm and 5370 ppm

The results are presented in Table. It was found that the gas-sensitive chip meets the specified parameters and provides sensitivity to detected gases up to 1 ppm.

Table

Chip characteristics

Isopropanol concentration	Sensitivity, ppm	Detection rate, s	T heater 210 °C
n	S	t	
1150	1.01	219	
1150	0.99	287	
5370	1.18	340	
5370	0.96	417	

Thus, due to the location of the heaters on the front side of the substrate and the high thermal conductivity of SiC, the gas-sensitive chip reaches the mode faster. Evaluation of the effect of an additional protective layer of SiO_2 demonstrated the absence of an effect of this film on gas sensitivity. For this purpose, samples with and without SiO_2 film were placed in an environment with detectable gas (isopropanol) at a temperature for 10 days and it was found that the deviations in resistance were insignificant.

Conclusion

Thus, the use of the proposed method for manufacturing a gas-sensitive multisensor chip allows to increase performance with higher temporal stability. It has been established that the protective SiO_2 layer does not impair the sensitivity of the gases being detected.

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