INVESTIGATIONS OF GAS-SENSITIVE NANOSIZE ZnO<Al> FILMS

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1. Introduction

For last few years the thin-film gas sensors made of metal oxide materials sensitive to different hazardous and explosive gases were subjects of intensive investigations [1-4]. Different technological methods were developed for manufacturing of thin film materials. The main disadvantage of the investigated thin-film gas sensors is the high working temperature [5-7]. The metal oxides have large band gap and insulator properties with high specific resistance (~10^{12} \ \text{Ohm.cm}). Therefore a notable change in their resistance (which is the basis of gas sensor functioning) is reachable only at high-enough heats. The aim of this work was the manufacturing and investigation of gas sensors made of nanosize ZnO<Al> films.

2. Experimental

The doping of the wide band gap oxide with an electroactive impurity was carried out for manufacturing of compositions with semiconductor properties and sufficient conductivity. In particular, compact targets with the diameter ~ 40 mm and thickness ~ 3 mm from ZnO<Al> were manufactured. The semiconductor ceramic samples ZnO, doped with 1 at.% aluminum were synthesized by the method of solid-phase reactions in air under the program: rise of temperature from room up to 800ºC for two hours, the soaking at this temperature during 30 minutes, the further increase in temperature for 2 hours prior to 1200ºC, the soaking at this temperature during 3 hours, and the subsequent slow cooling. Obtained ZnO<Al> samples had the specific conductivity ~ 5\times10^{-4} \ \text{Ohm}^{-1}.\text{cm}^{-1}. The produced targets are intended for the manufacture of thin films on glassceramic substrates by the magnetron sputtering method. The power of the magnetron was 80 W, and the duration of the magnetron sputtering changed from 30 to 60 minutes. The modes of sputtering were chosen in a way to provide the manufacturing of homogeneous films. Platinum layer (which, as is known, has catalitic and selective properties)
and then gold interdigitated ohmic contacts were evaporated on prepared films by the ion-beam sputtering.

The thickness of prepared films was measured using Ambios XP-1 Stylus Profiler. Investigations of the surface of films were carried out using SEM Vega 5130 MM (TESCAN).

Five percent of H$_2$ in N$_2$ premixed gas was used in the sensitivity examination. Before the gas entrance into the measuring chamber, it passed through a moisture trap. The sensitivity of obtained structures was examined at the gas 5000 ppm concentration. The process was controlled by a computer in the following way: PC keeps open the gas valve until the gas concentration in the testing chamber reaches the desired value. The measurements were carried out at different temperatures of heating of the working body (from room temperature up to 100$^\circ$C). All measurements were performed via computer control of the system.

3. Results and discussion

The thickness of films was equal to ~ 50-80 nm. The SEM analysis shows that the obtained ZnO<Al> thin films have typical grain sizes of ~ 20-30 nm. The SEM image of prepared ZnO<Al> films is shown in Fig. 1.

![SEM image of ZnO<Al> films](image)

Fig. 1. The SEM image of ZnO<Al> films.

Results of investigations of the sensitivity of the obtained glassceramic/ZnO<Al> structures to hydrogen gas at gas concentration of about 5000 ppm at different temperatures of the working body are presented in Fig. 2. These results show that glassceramic/ZnO<Al> structures can work at low temperatures. Resistance of the sensor decreases ~ 8 times at gas supply by heating up to 60$^\circ$C. The response time of the system is 6-7 minutes at hydrogen supply by heating a working
body up to 50°C. This time decreases with increase in the heating temperature, reaching 2.5 minutes by heating up to 100°C; thus, the basic change of resistance takes place during the first 1.5 minutes. Peculiarity of the structure glassceramic/ZnO<Al> is a small recovery time of the system: it is ~ 1.5-2.5 minutes and practically does not depend on temperature of the working body heating. The recovery time of the system is less than the response time at the heating of the working body up to 80°C. Thus, we can notice that, for recovering of the system, no additional actions (such as, for example, often used, but undesirable, pulse heating to the temperature exceeding several times the temperature of the working body) are necessary.

The change in duration of the magnetron sputtering (from 30 up to 60 minutes) allowed the manufacturing of films with different thickness. The results of investigations of the influence of hydrogen on the resistance of films with different thickness are presented in Fig. 3. We obtained that films manufactured for 30-50 minutes had almost identical sensitivity (resistance of the structure decreases ~ 4 times), the response time of the system was ~ 4-5 minutes, and the recovery time was ~ 2 minutes. An increase in the duration of magnetron sputtering up to 60

![Fig. 2. Sensitivity of glassceramic/ZnO<Al> structure (duration of sputtering 30 minutes) to 5000 ppm hydrogen at different temperatures.](image2)

![Fig. 3. Resistivity of glassceramic/ZnO<Al> structure at 5000 ppm of hydrogen at 100°C (duration of the magnetron sputtering is indicated).](image3)
minutes leads to the increase in the sensitivity (resistance of structure decreases in ~ 8 times), however, the recovery time increases up to ~ 5 minutes (the response time was ~ 4 minutes).

4. Conclusion

Thus, we have manufactured ZnO<Al> thin films on glassceramic substrates by the magnetron sputtering method. The obtained ZnO<Al> thin films have nano-size grains (~ 20-30 nm), the thickness of films was equal to ~ 50-80 nm. The glassceramic/ZnO<Al> structure shows sufficient sensitivity to hydrogen at heating of a working body already up to 60°C. The obtained structure satisfies the basic demands in making low temperature to gas sensor today. The developed sensor has small response and recovery times, rather low working temperature, and small sizes. Such structures can be used in manufacturing of single hydrogen sensor and “electronic nose” as well.

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REFERENCES