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Nanostructured Sensors for Detection of Hydrogen Peroxide Vapours

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Abstract: Solid-state sensors made from doped metal oxide ZnO<La> and SnO_2<Co> were prepared for detection of hydrogen peroxide vapours. Gas sensitive nanostructured films were manufactured by the high-frequency magnetron sputtering method. Thicknesses of deposited semiconductor nanostructured films were measured and its morphology was investigated. The average size of nanoparticles was equal to 18.7 nm for deposited films. The response of the prepared sensors was measured at different temperatures of the sensor work body and different concentrations of hydrogen peroxide vapours. It was found that both La-doped ZnO and Co-doped SnO_2 sensors exhibit a sufficient sensitivity to 10 ppm of hydrogen peroxide vapours at the operating temperature 220 °C and 200 °C, respectively. It was established that the dependencies of the sensor sensitivity on hydrogen peroxide vapours concentration at the work body temperature 150 °C have a linear characteristic for both prepared structures and can be used for determination of hydrogen peroxide vapours concentration.

Keywords: Sensor, Hydrogen peroxide vapours, Semiconductor, Nanostructured film, Doped metal oxide.

1. Introduction

Hydrogen peroxide (H_2O_2) is a chemical widely used in such fields as medicine, pharmacology, food and textile industry due to a wide spectrum of its antibacterial properties, low toxicity and ecological purity (it decomposes to produce neutral water and oxygen). However, pure H_2O_2 at large concentrations is explosive under certain conditions (for example, in the presence of transition metals). Therefore, concentrated solutions of H_2O_2 can cause burns in the case of the contact with skin, mucous membranes and respiratory tract. H_2O_2 is subsumed under the category of matters those are dangerous for man with certain maximum permissible concentration. Therefore, the development of sensors for determination of the H_2O_2 concentration in the environment is important and attracts interest of chemists, physicians, industrial engineers, etc. The H_2O_2 stable sensors can be used in analytical chemistry, in various fields of the industry

(food, wood pulp textile, pharmacology), for the environmental control, in clinical diagnostic for prompt and reliable specification of diagnoses of different diseases and checking of a course of treatment.

Several techniques have been developed for a reliable and sensitive determination of H_2O_2, such as chemiluminescence, spectrophotometry, fluorimetric and colorimetric detection, liquid chromatography, electroanalytical and optical interferometry [1-5]. These techniques are complex, expensive and time consuming. Now the electrochemical sensors are used [4-9]. A large range of materials such as ferric hexacyanoferrate (Prussian blue) and other metal hexacyanoferrates, metallophthalocyanines and metalloporphyrins, transition metals and metal oxides have been employed for the manufacture of these sensors. The advantages of these sensors are simplicity of manufacturing, good response and capability of control in a real time. In recent years, nanotechnology progress is promoted advance in the field of manufacturing of the H_2O_2 electrochemical sensors. For example, carbon nanotubes and graphene can be used either as substrates with high specific area for catalytic materials or as electrocatalysts by themselves [10-13].

H_2O_2 serves as a disinfectant for medical equipment and surfaces as well as for sterilizing surgical instruments. Therefore, the correct selection of the H_2O_2 concentration during the sterilization of the equipment technological surfaces and also control of the H_2O_2 content in air after completion of disinfection cycle are very important. Note that the process of chemical decontamination can be carried out in two different ways: the first one is the wet approach using water or any other solutions of H_2O_2 (certain concentration) and the second one is the dry method using H_2O_2 in vapour phase [14]. Therefore, the development and manufacturing of stable and reproducible sensors sensitive to H_2O_2 vapours are extremely required [15-17]. The checking of H_2O_2 vapours phase is also crucially significant in connection with counterterrorism efforts. The sensors sensitive to H_2O_2 vapours may find application in the detection of peroxide-based explosives [18, 19].

The most used method is based on the determination of the concentration of H_2O_2 vapours after cooling down and being absorbed in the water. The near infrared spectrophotometry was used for the monitoring of the concentration of H_2O_2 vapours in the course of sterilization [20]. The chemiresistive films made from organic p-type semiconducting phthalocyanines metalized with elements of p-, d-, and f-blocks were sensitive to H_2O_2vapours [18]. An amperometric sensor for detection of H_2O_2 vapours made of an agarose-coated Prussian-blue modified thick-film screen-printed carbon-electrode transducer was investigated [21]. It was reported about organic single-wire optical sensor for H_2O_2 vapours made of organic core/sheath nanowires with wave guiding core and chemiluminogenic cladding [22].

The aim of the present paper is development of technology, manufacture and investigation of solid-state H_2O_2 vapours sensors made from semiconductor metal oxide nanostructured films.

Here, the necessities in the development of the H_2O_2 sensors and, in particular, sensors sensitive to H_2O_2 vapours are briefly described. The manufacturing technology of sensors made from semiconductor doped with metal oxide ZnO<La> and SnO_2<Co> nanostructures is given in the second section. The results of the investigations of prepared sensors sensitivity to H_2O_2 vapours are presented in the third section. In the fourth section, the conclusions are made and the directions of the future work are described.

2. Experiments

Ceramic targets made from metal oxide ZnO doped with 1 at.% La or SnO_2 doped with 2 at.% Co were synthesized by the method of solid-phase reaction in the air into the programmable furnace Nabertherm, HT O4/16 with the controller C 42. The following program of annealing for the compact samples of ZnO<La> was chosen: rise of temperature from room temperature up to 1300 °C for three hours, soaking at this temperature during four hours, further decrease in the temperature for three hours prior to room temperature. The annealing of the compacted samples SnO_2<Co> was carried out at 500 °C, 700 °C, 1000 °C and 1100 °C consecutively, soaking at each temperature during five hours. Then, the synthesized compositions were subjected to mechanical treatment in the air in order to eliminate surface defects. Thus, smooth, parallel targets with a diameter ~ 40 mm and thickness ~2 mm were manufactured.

The prepared ZnO<La> and SnO_2<Co> targets had sufficient conductance and were used for deposition of nanosize films. Multi-Sensor-Platforms (purchased from TESLA BLATNÁ, Czech Republic) are used as substrates. Chips can be kept at constant temperature using heat resistance. The platform integrates a temperature sensor (Pt 1000), a heater and interdigitated electrode structures with platinum thin film on a ceramic substrate. Heater and the temperature sensor are covered with an insulating glass layer. Gas sensitive layer made from ZnO doped with 1 at.% La or SnO_2 doped with 2 at.% Co was deposited onto the non-passivated electrode structures using the high-frequency magnetron sputtering method. The Multi-Sensor-Platforms are converted into gas sensors that way.

The following working conditions of the high-frequency magnetron sputtering were chosen: the power of the magnetron generator unit was equal to 60 W; the substrate temperature during sputtering was equal to 200 °C; the distance between substrate and target was equal to 7 cm; the duration of the sputtering process was equal to 15 minutes and 20 minutes for ZnO<La> and SnO_2<Co>, respectively. The sensing device was completed through the ion-beam sputtering

deposition of palladium catalytic particles (the deposition time ~ 3 seconds). Further annealing of the manufactured structures in the air was carried out at temperature 250 °C to obtain homogeneous films and eliminate mechanical stresses.

The thicknesses of the deposited doped metal oxide films were measured by Ambios XP-1 profilometer. The morphology and chemical composition of the deposited SnO_2<Co> and ZnO<La> films were studied by scanning electron microscopy (SEM) using Mira 3 LMH (Tescan) and energy-dispersive X-ray spectroscopy using Quantax 200 with XFlash 6|10 detector (Bruker) with resolution of 127 eV, respectively.

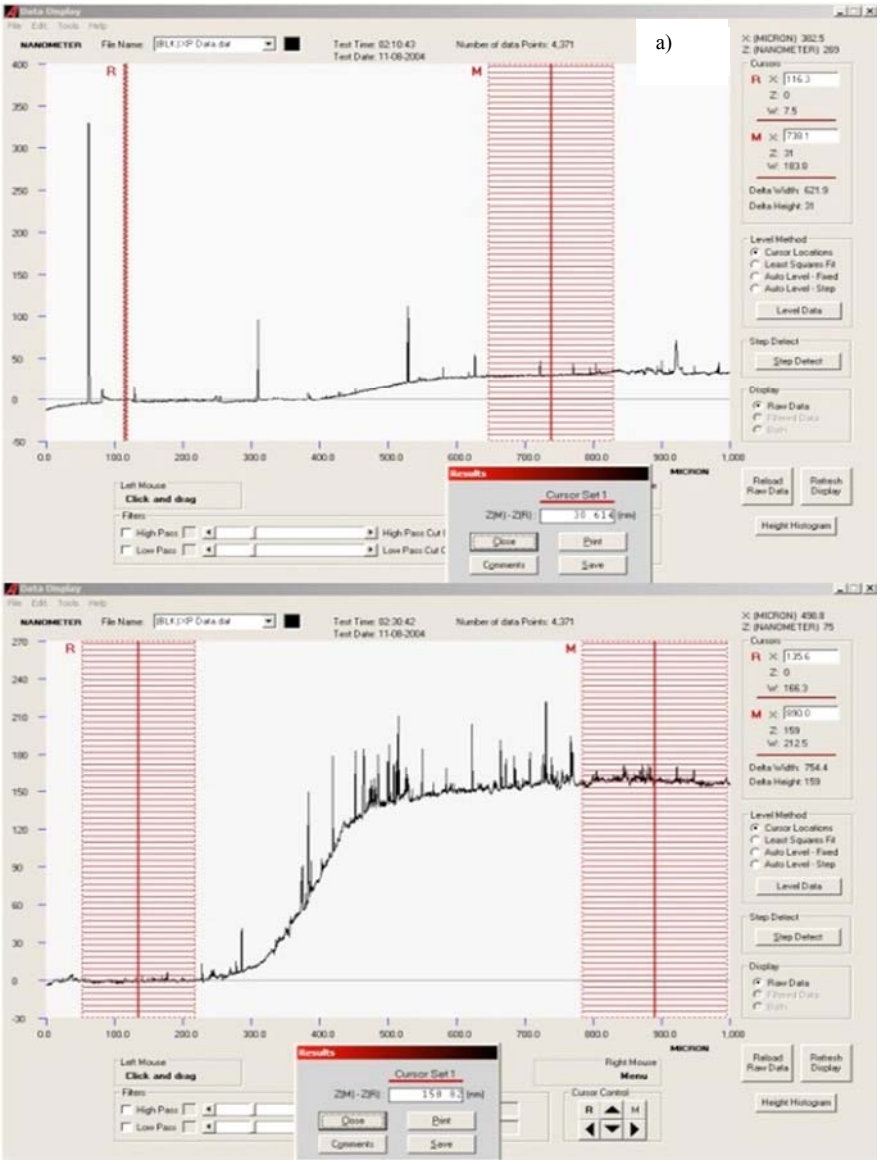

Fig. 1. The thicknesses measurements resultes for ZnO<La> (a) and SnO_2<Co> (b) films.

The gas sensing properties of the prepared sensors made from doped metal oxide films under the influence of H_2O_2 vapours were measured at Yerevan State University using a home-made developed system (see, for example, [23]). The sensors were placed in a hermetic chamber. A certain quantity of H_2O_2 water solution was placed in the chamber to reach a corresponding concentration of H_2O_2 vapours. The measurements of the manufactured sensors response (the sensor resistance changes under the H_2O_2 vapours influence) were carried out at different concentrations of H_2O_2 vapours (from 100 ppm up to 4000 ppm). The platinum heater on a front side of the sensor ensures a necessary temperature of the work body. The sensor work body temperature was varied from room temperature up to 350 °C. All measurements were carried out at the sensor applied voltage of 0.5 V. As a result of such measurements, the sensor sensitivity was determined as the ratio $R_{peroxide}/R_{air}$, where $R_{peroxide}$ is the sensor resistance in the presence of H_2O_2 vapours in the air and R_{air} is the sensor resistance in the air without H_2O_2 vapours.

The investigations of the sensitivity of the prepared sensors to H_2O_2 vapours with concentration lower than 100 ppm were carried out at University of Chemistry and Technology (Prague). In particular, the measurements of the response of the ZnO<La> sensors to 10 ppm H_2O_2 vapours were carried out by the following way. Firstly, an atmosphere containing 10 ppm of H_2O_2 vapours was prepared in a laboratory model of an isolator. This H_2O_2 vapours concentration decreased by spontaneous decomposition of H_2O_2. When a reference device (Dräger Sensor® H_2O_2 HC) could not detect any H_2O_2 vapours, the ZnO<La> sensor was inserted into the model isolator. Then, sensor responded immediately. When the maximum response was reached, the sensor was taken out into an atmosphere without any traces of H_2O_2 vapours. This process was repeated three times. The measurements of the response to 75 ppm H_2O_2 vapours were carried out for the SnO_2<Co> sensors in the same way.

The temperature dependence of sensitivity to 10 ppm H_2O_2 vapours were investigated for the SnO_2<Co> sensors. For these measurements, atmosphere in "Peroxybox" system, developed in the same Institute in Prague, was controlled (0-10 ppm H_2O_2 and 20-23% RH) and the sensor's temperature was changed. The final sensitivity was calculated as the response of sensor in "Peroxybox" system divided by the response of sensor in the air.

The temperature dependence of sensitivity to 100 ppm H_2O_2 vapours were investigated in the same way for the ZnO<La> sensors.

3. Results and Discussion

The thicknesses of the ZnO doped with 1 at.% La and SnO_2 doped with 2 at.% Co films were equal to 30 nm and 160 nm, respectively (Fig. 1).

The results of the study of morphology for the deposited doped metal oxide films are presented in Fig. 2. The average size of nanoparticles was equal to 18.7 nm for both compositions.

The sensors manufactured by us are resistive. The operation of this type of sensors grounds on the changes in the electrical resistance of gas sensitive semiconductor layer under the influence of H_2O_2 vapours due to an exchange of charges between molecules of the semiconductor film and adsorbed H_2O_2 vapours.

The H_2O_2 decomposes to produce water vapours and oxygen:

$$2H_2O_2 \rightarrow 2H_2O\uparrow + O_2\uparrow . \quad (1)$$

These adsorbed oxygen molecules capture the electrons from the semiconductor film:

$$O2+ e- \rightarrow O2- . \quad (2)$$

A variation of the sensor resistance takes place as a result of such exchange byelectrons. This variation of the resistance was fixed as a sensor response.

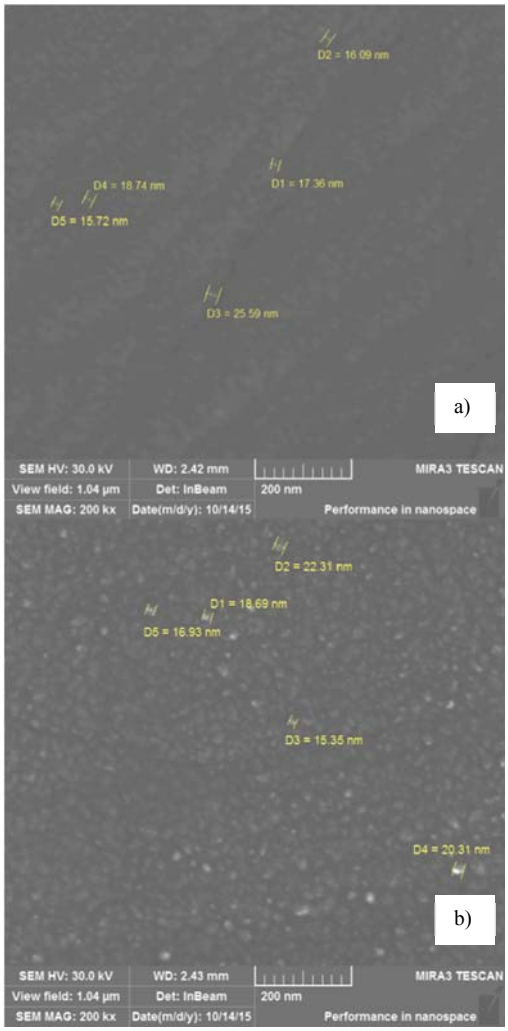

Fig. 2. The SEM images for SnO_2 doped with 2 at.% Co (a) and ZnO doped with 1 at.% La (b) films.

The sensor resistance variation under the H_2O_2 vapours influence was measured at Yerevan State University using the static gas sensor test program controlled a special home-made computer system. Typical curve demonstrating the change in the sensor resistance under the influence of H_2O_2 vapours at invariable temperature of the work body is presented in Fig. 3a. As mentioned above, the sensitivity of sensors was calculated as the ratio $R_{peroxide}/R_{air}$, where $R_{peroxide}$ and R_{air} are the sensor electrical resistances in the H_2O_2 vapours atmosphere and in the air, respectively. The ZnO<La> sensor sensitivity at different work body temperatures is presented in Fig. 3b.The response and recovery times were determined as the time required for reaching the 90% resistance changes from the corresponding steady-state value of each signal. For the ZnO<La> sensors the response and recovery times were equal to 6-8 and 10-12 minutes in average, respectively, at the temperatures more than 200 °C.

The results of such investigations for SnO_2<Co> sensors are presented in Fig. 4. For this structure both the response and recovery times were equal to 5 minutes at the temperatures more than 200 °C.

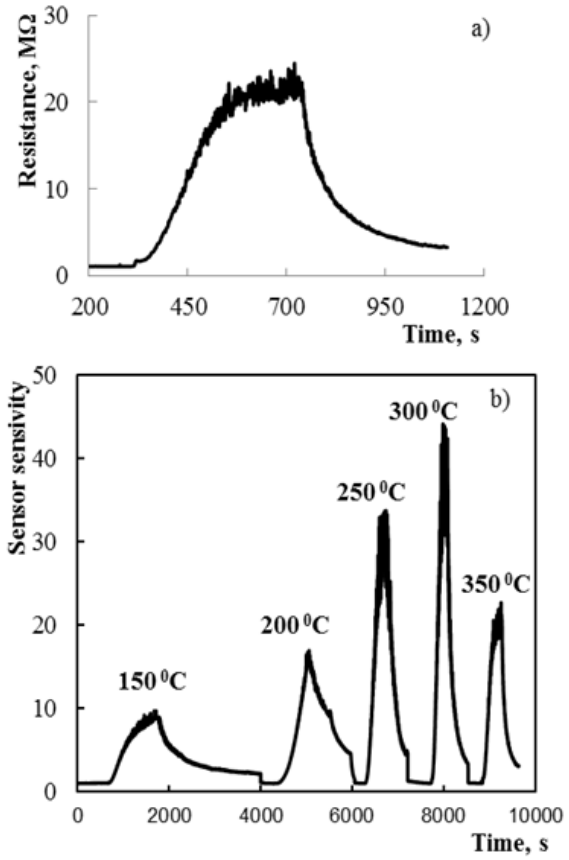

Fig. 3. a)The resistance change of the ZnO<La> sensor under the influence 1800 ppm of H_2O_2 vapours, work body temperature 350 °C;
b) The sensitivity to 1800 ppm of H_2O_2 vapours for ZnO<La> sensor at different work body temperatures.

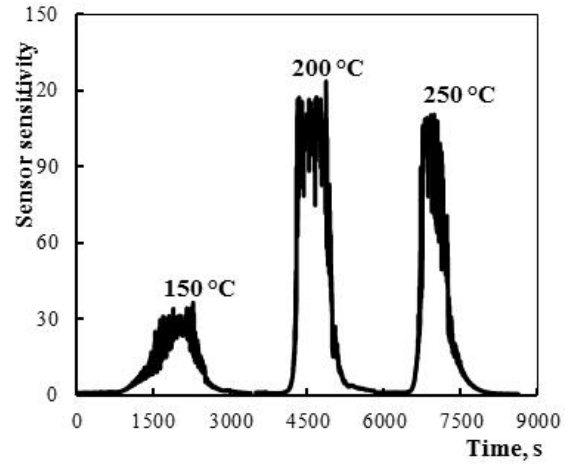

Fig. 4. The sensitivity to 100 ppm of H_2O_2 vapours for SnO_2<Co> sensor at different work body temperatures.

As shown in Fig. 3b and Fig. 4, the sensitivity of the sensors decreases for both structures, when the working body temperature excesses some certain value (300 °C and 250 °C for ZnO<La> and SnO_2<Co> sensors, respectively). The amount of vapour molecules adsorbed on a surface and generally held by Van der Waals forces (physical adsorption), decreases with the increasing of temperature. More intensive exchange of electrons between the absorber and the absorbed molecules takes place when the stronger chemical nature bond is established between its, originated at capping of electronic shells of both adsorbent and adsorbate atoms. The amount of chemisorbed centers increases with increasing the temperature. The desorption prevails over the adsorption when a temperature is increased above certain value and, therefore, the sensor sensitivity decreases. The temperature, above which the sensitivity decreasing occurs, for the sensors made of ZnO<La> structure is greater than for the sensors made of SnO_2<Co>structure. Probably, the chemical bonds between molecules of ZnO and H_2O_2 are stronger than that of between molecules of SnO_2 and H_2O_2. This is also testified by the fact that the recovery time for sensors made of SnO_2<Co> is less than that of for ZnO<La> sensors.

Note, the prepared sensors resistance has changed in order of magnitude under influence of H_2O_2vapours starting at operation temperature of 100 °C. However, a longer time was needed for recovery of the sensors parameters at such temperature. The pulsed increasing in the work body temperature is needed for decreasing of the recovery time of the investigated sensors.

As it has already been noticed, the H_2O_2 belongs to the type of materials dangerous for man with certain maximum permissible concentration. The permissible limit of exposure of 1,0 ppm has established by Occupational Safety and Health Administration (OSHA, USA) [16, 20]. It is immediately dangerous for life and health when its concentration reaches 75 ppm [3]. Therefore, the investigations of the prepared SnO_2<Co> and ZnO<La> sensors gas sensing properties to H_2O_2 vapours with concentration lower than 100 ppm were also carried out at University of Chemistry and Technology (Prague).

The results of these investigations are presented in Fig. 5 and Fig. 6. The investigations of the sensors sensitivity to very low concentrations (0-10 ppm) of H_2O_2 vapours show, that the structure made of SnO_2<Co> exhibits a response to 10 ppm of H_2O_2 vapours at the operating temperature starting at 50 °C (Fig. 6b). The sensitivity to 10 ppm of H_2O_2vapours was equal to ~ 3 for the SnO_2<Co> sensors at the work body temperature of 200 °C. The sensitivity to 10 ppm of H_2O_2 vapours was equal to ~ 2 for the ZnO<La> sensors at the work body temperature of 220 °C. Note that the DrägerSensor® H_2O_2 HC reference device is not sensitive to 10 ppm of H_2O_2vapours (Fig. 5a). These measurements were carried out at University of Chemistry and Technology (Prague).
The results of investigations of the prepared sensors sensitivity at different concentrations of H_2O_2 vapours are presented in Fig. 7.

As can see, these dependencies sensitivity on H_2O_2vapours concentration have a linear characteristic for both type of sensors and can be used for determination of H_2O_2 vapours concentration.

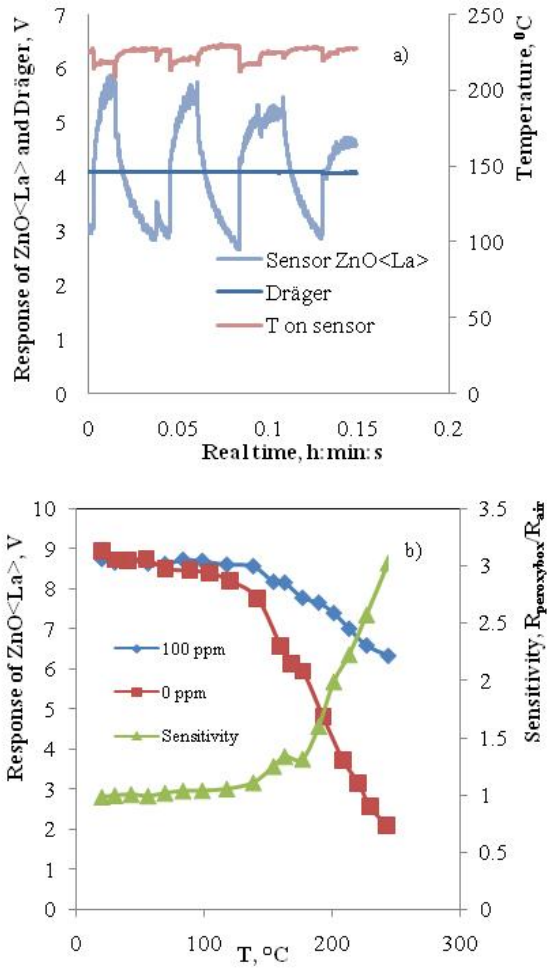

Fig. 5. The response to 10 ppm of H_2O_2 vapours at the operation temperature of 220°C (a) and the temperature dependence of sensitivity to 100 ppm H_2O_2 vapours (b) for ZnO<La> sensors.

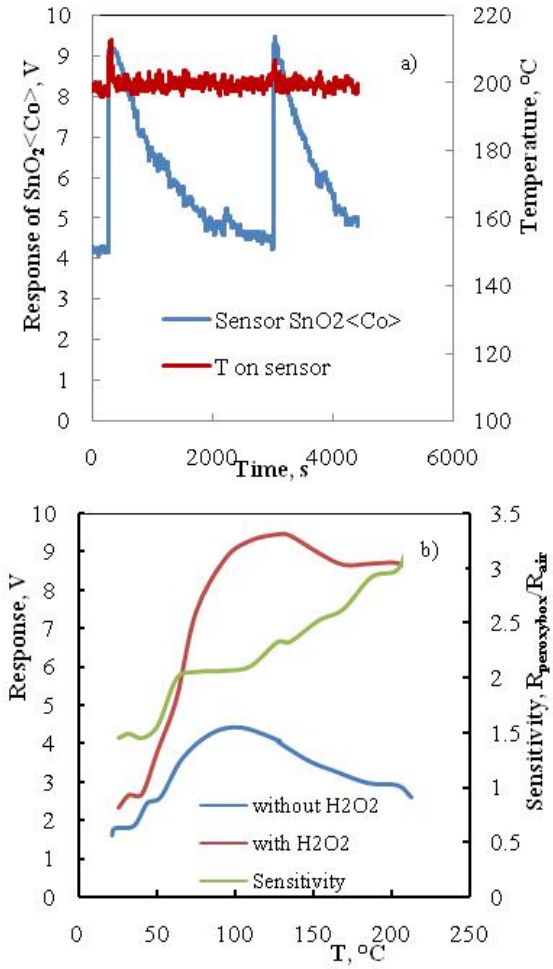

Fig. 6. The response to 75 ppm of H_2O_2 vapours at the operation temperature of 200°C (a) and the temperature dependence of sensitivity to 10 ppm H_2O_2 vapours (b) for SnO_2<Co> sensors.

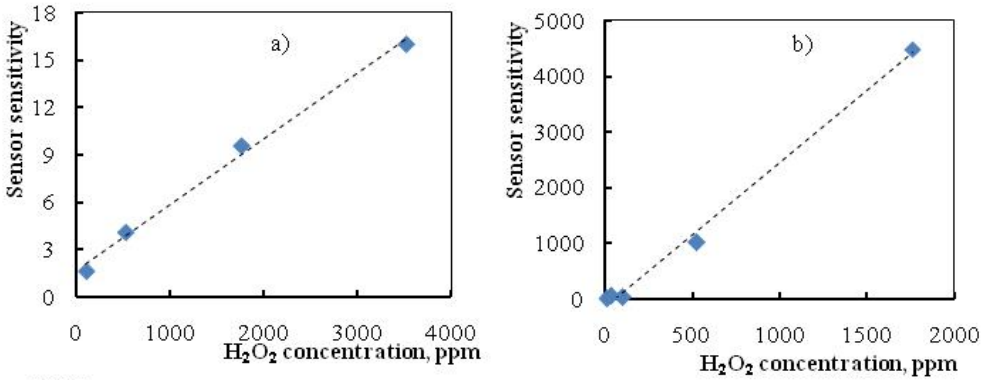

Fig. 7. The dependence of the sensitivity on H_2O_2 vapours concentration at operating temperature of 150 °C for ZnO<La> (a) and SnO_2<Co> (b) sensors.

7. Conclusions

The technology for the manufacturing of the semiconductor sensors made from ZnO doped with 1 at.% La and SnO_2 doped with 2 at.% Co nanostructured films was developed. The gas sensitive ZnO<La> and SnO_2<Co> layers were deposited onto the Multi-Sensor-Platforms using the high-frequency magnetron sputtering method. The thicknesses of the deposited doped metal oxide films were equal to 30 nm and 160 nm for ZnO<La> and SnO_2<Co>compositions, respectively. The average size of the nanoparticles was equal to 18.7 nm for both structures. Test specimens detecting H_2O_2 vapours

were manufactured and investigated. The sensitivity of the prepared sensors was measured at different work body temperatures of the sensor and concentrations of H_2O_2vapours. It was found that both Co-doped SnO_2 and La-doped ZnO sensors exhibit a good response to H_2O_2 vapours at the operating temperature starting from 100 °C. The sensors made from SnO_2<Co> and ZnO<La> were sufficiently sensitive to 10 ppm of H_2O_2 vapours. It was established that the dependencies of the sensitivity on H_2O_2 vapours concentration at the work body temperature 150 °C have a linear characteristic for prepared structured and can be used for determination of H_2O_2 vapours concentration. Our future work will be directed to the long-time stabilization of sensors parameters and the improvements of such characteristics as operation speed and recovery time.

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