

The ethanol sensors made from α -Fe₂O₃ decorated with multiwall carbon nanotubes

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Abstract. Thin film ethanol sensors made from α -Fe₂O₃ decorated with multiwall carbon nanotubes (MWCNTs) were manufactured by the electron beam deposition method. The morphology of the decorated α -Fe₂O₃/MWCNTs (25:1 weight ratios) nanocomposite powder was investigated using the scanning electron microscopy and X-ray diffraction techniques. The thickness of thin films has been determined from ellipsometric measurements. The response of manufactured sensors was investigated at different temperatures of the sensor work body and concentration of gas vapors. Good response of prepared sensors to ethanol vapors already at work body temperature of 150°C was shown.

Keywords: gas sensor; Fe₂O₃; carbon nanotubes; response; ethanol

1. Introduction

An interest to application of portable alcohol sensors is caused by possibilities of their use in modern technics medicine, biotechnology etc. In particular, sensors can be used also for control of different chemical processes and food quality, determining of a level of alcohol in wines and a degree of human intoxication. It is very important to detect the gas vapors and measure its concentration, toxicological and psychological effects in the atmosphere and different environments (Vrnata *et al.* 2000, Ling *et al.* 2006, Xu *et al.* 2008, Zhang *et al.* 2008, Prajapati *et al.* 2011, Beckers *et al.* 2013).

As usually, all of the most important parameters of the semiconducting resistive gas sensors depend on the correct choice of its sensing material. Different metal oxides are mainly used as sensitive materials. It is known that metal oxides are very sensitive to a composition of

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environment (see, for example, Aroutiounian 2013b). Pure α -Fe₂O₃ is quite inert material, it weakly interact with gases. But, in particular, thin and thick films made of α -Fe₂O₃ doped with different impurities or catalytic metal particles are rather sensitive to alcohol vapors (Tan *et al.* 2000, Ivanovskaya *et al.* 2003, Neri *et al.* 2005, Pawar *et al.* 2012). A correct choice of impurities doped leads to not only to an improvement in the response to a target gas, other important parameters like the long-term stability, operation speed etc.

Note that gas sensors made from nanostructures (nanotubes, nanowires, nanoparticles) have a promising future because of their very large surface-to-volume ratio and better adsorption of gas molecules. From this point of view, multiwall carbon nanotubes (MWCNTs) are usable nanomaterials in modern science and engineering. They are characterized by the high mechanical durability, flexibility, chemical and long-time stability. This number of features became the base for investigation of possible use of MWCNTs during the manufacture of gas sensors. They offer a high potential for gas sensing devices. However, the lack of the selectivity, long response and recovery times as well as low response limit the practical application of pristine MWCNTs.

There are several methods to functionalize of MWCNTs. For example, catalytic metal nanoparticles are frequently incorporated on the surface of MWCNTs acting as active sites during the interaction between target analytes and MWCNTs. It improves their sensing properties. The sensor device consisted on a film of sensitive material (MWCNTs-Pd) deposited by drop coating on platinum inter-digitated electrodes (Colindres *et al.* 2014). The sensor exhibited a change of the resistance in contact with ozone (O₃) and had the response time about 60 s at different temperatures, the capability of detecting concentrations up to 20 ppb, high repeatability, full recovery and rather efficient response. The best response was found for the sensor operated at the work temperature 120°C. Other authors reported response time between 100 to 1200 s during the ozone detection. Nanoclusters of Co catalysts were sputtered on the surface of the MWCNT film to improve NH₃ gas sensitivity in comparison to pristine unfunctionalized MWCNT films. The NH₃ sensitivity of Co-functionalized MWCNT-based gas sensors was detected 7-28 ppm NH₃ at room temperature (Nguyen *et al.* 2013).

Very important is a possibility to prepare metal oxide/MWCNT samples which are sensitive to different gases. Nanocomposites based on Pt-doped TiO₂/MWCNTs have been introduced as the H₂ sensitive materials at low temperatures (Trocino *et al.* 2012). MWCNTs embedding in the TiO₂ matrix modify markedly the electrical conductivity (the resistance of the sensing layer decreased). Pt acted as a catalytic additive. Pt-TiO₂/MWCNTs-based sensors were found to be sensitive to hydrogen at concentrations between 0.5 and 3% in air, satisfying the request for practical applications in hydrogen leak detection devices. The results obtained suggest then that the sensing performance of the hybrid nanostructure sensor could be attributed to: (i) the increase in the specific surface area of hybrid nanostructures; (ii) the effective electron transfer between TiO₂ grains and MWCNTs; (iii) the catalytic action of platinum.

We established earlier that the addition of MWCNTs into the SnO₂ metal oxide allowed improving the long-time stability of the sensor parameters, increasing the operation speed, as well as decreasing in the work body temperature of the sensor (Aroutiounian *et al.* 2012, Aroutiounian *et al.* 2013a). It is possible even detect isobutane using such a sensor. That is impossible by CNTs and SnO₂ separately, i.e., we have a synergistic effect in this case. Important experiments with different metal oxides, carbon nanostructures and/or different preparation procedures are in progress in order to better understand the sensing properties of these composite materials and fabricate gas sensors with enhanced performances.

In this work, we reported about the manufacture and investigations of resistive thin film ethanol

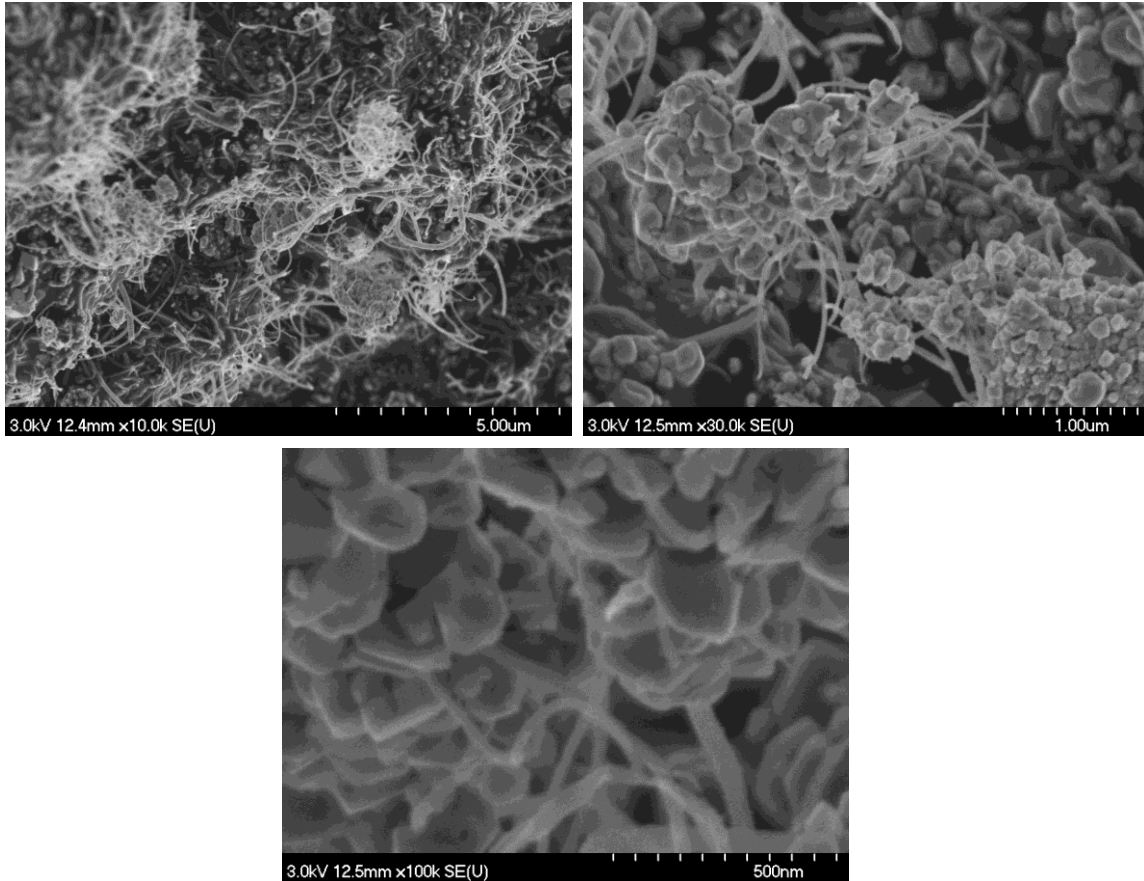


Fig. 1 SEM images with different magnification for decorated α -Fe₂O₃/MWCNTs (25:1) nanopowder

sensors made from α -Fe₂O₃ decorated with MWCNTs with weight ratios 25:1. Possibility of combined use of MWCNTs with metal oxide compositions as complete gas sensitivity systems was investigated. Results of measurements of the sensors response to ethanol vapors were analyzed. We use standard techniques for manufacture of gas sensors and measurements of the work body temperature (see for example Aroutiounian *et al.* 2012, Aroutiounian *et al.* 2013a).

2. Material and measurements methods

The decorated α -Fe₂O₃/MWCNTs (25:1) nanocomposite powder was manufactured by researchers from University of Szeged (Hungary). The MWCNTs were functionalized in a simple method: the tubes were continuously stirred in cc. HNO₃ for 3 h and after that the mixture was filtrated and the MWCNTs were washed with distilled water until neutral pH was reached. The α -Fe₂O₃/MWCNTs composite was prepared by an impregnation method. The MWCNTs was added in distilled water and a suspension was prepared via sonication for 45 min. During this time, the calculated amount of precursor was dissolved in another portion of distilled water. It was continuously stirred for complete dissolution during half hour using a magnetic stirrer. Finally, the

solution of the precursor was added drop by drop into the MWCNTs-suspension and then the mixture was heated to 90°C to evaporate water on a heated magnetic stirrer. When the solvent was completely removed, a reddish black powder was obtained. Further this powder was dried at 110°C for 24 h, and then it was annealed in air for 3 h at 400°C.

The morphology of the decorated α -Fe₂O₃/MWCNTs (25:1) nanocomposite powder was investigated by scanning electron microscopy (SEM). The SEM analysis was carried out on a Hitachi S-4700 Type II FE-SEM microscope equipped with a cold field emission gun operating in the range of 5-15 kV. The samples were mounted on a conductive carbon tape. SEM images with different magnification for decorated α -Fe₂O₃/MWCNTs (25:1) nanocomposite powder are presented in Fig. 1. As one can see, the MWCNTs diameter was equal approximately 30-40 nm, size of α -Fe₂O₃ particle was made average ~100-110 nm. It could be an advantage in view of the gas sensation that the size of the corners and ledges of Fe₂O₃ particles are also in nanometric scale. MWCNTs were wrapped by grains of α -Fe₂O₃, thus a part of carbon nanotubes remains uncover. Our opinion is that this composite material is not just a mechanical mixture of the components. This idea is based on the results of an ultrasonic treatment in ethanol which had no effect on the structure and morphology of the nanocomposite. The co-authors also described in an earlier publication (Berki *et al.* 2013) that chemical bond can be revealed with infrared spectroscopy between the carbon nanotubes and inorganic covering materials. We suppose the presence of this bond in case of this nanocomposite too. It is possible to suggest that both MWCNTs and α -Fe₂O₃ will made the contribution to sensor response at such structure of nanocomposite.

Powder XRD analysis was effectuated on a Rigaku Miniflex-II Diffractometer (angle-range: $\theta=20-70^\circ$) utilizing characteristic X-ray (CuK α) radiation. Results of XRD analysis for α -Fe₂O₃/MWCNTs (25:1) nanocomposite powder are presented in Fig. 2. The Miller indexes belong

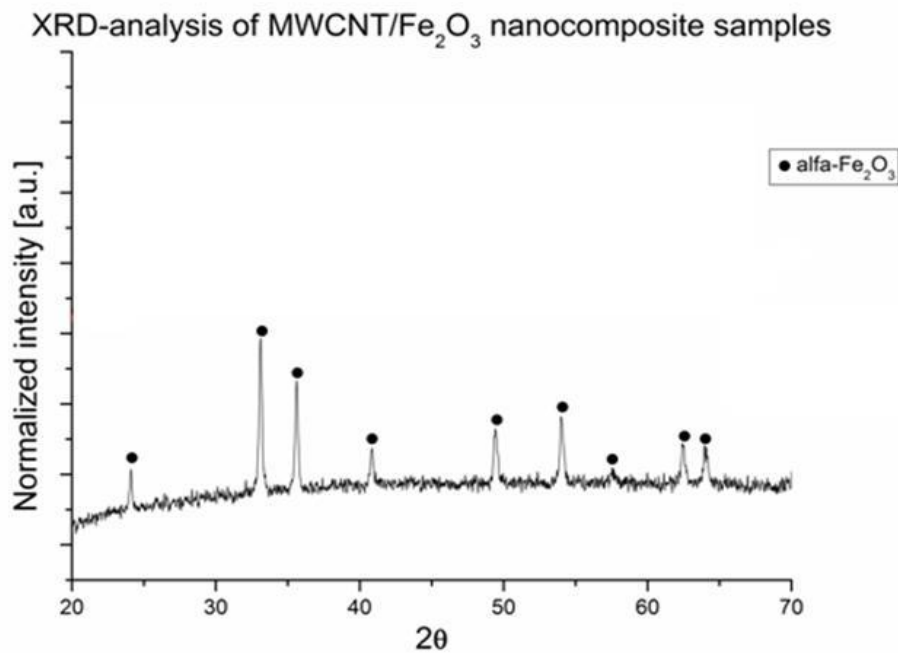


Fig. 2 The XRD spectra of the decorated α -Fe₂O₃/MWCNTs (25:1) nanopowder

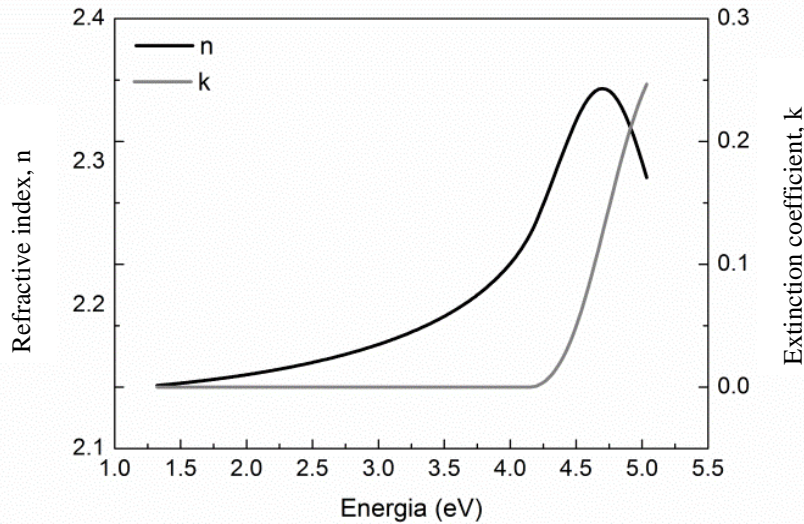


Fig. 3 Optical data from ellipsometric measurements for sensors made from α -Fe₂O₃ decorated with MWCNTs. Data about refractive index (n) is given on Fig. 3 on the left y-axis and extinction coefficient (k) – on the right y-axis

to the reflections of α -Fe₂O₃: (012), (104), (110), (113), (024), (116), (122), (214) and (300). Due to their mass ratio reflexion characteristic for MWCNT at $2\theta=26.1^\circ$ cannot be detected.

Thin films made of nanocomposite powder above mentioned were prepared. First, 3 weight % of polypropylene as binder was added to the decorated α -Fe₂O₃/MWCNTs (25:1) nanopowder. Then, tablets with diameter of 3 mm and height 4 mm were pressed at 160°C from the obtained mix. The prepared tablets were used as a target for the electron beam evaporation of thin films. Optimal parameters of the evaporation were found: polycore substrate temperature was equal to 200°C, distance between substrate and target - 5 cm, evaporation time - 15 min.

Further, for the manufacture of sensors, palladium catalytic nanoparticles and gold inter-digital Ohmic contacts were realized on the surface of all prepared structures using the ion-plasma sputtering method. The duration of palladium deposition process was equal to 2 seconds. Gold was deposited within 1 hour. The subsequent annealing of obtained samples in the air was carried out at 350°C during 2 h for the improving in the adhesion of contacts, minimization of any possible mechanical stresses and stabilization of sensors parameters. In result, the sensors made from α -Fe₂O₃ decorated with MWCNTs with weight ratios 25:1 were manufactured by us.

Ellipsometric measurements (Woollam M-2000F rotating compensator spectroscopic ellipsometer) were carried out for to measure the thickness of the layers. Usually, spectroscopic ellipsometry is widely used to determine optical properties and layer thickness of thin film samples. Ellipsometry is based on that the polarization state of light changes upon reflection on a sample. This change is in correlation with optical properties (refractive index - n , and extinction coefficient - k) and the thickness of the layer. Spectroscopic ellipsometry measures the ratio of the Fresnel reflection coefficients of the two components of the light parallel (p) and perpendicular (s) to the plane of incidence according to the following equation

$$\rho = \frac{r_p}{r_s} = \text{tg}\Psi e^{i\Delta} \quad (1)$$

where Ψ and Δ are the ellipsometric angles being Ψ the amplitude ratio and Δ the phase difference between the p and s components. Evaluation requires an optical model and the n , k and thickness values can be derived from the measured data with an indirect fitting procedure. The Ψ and Δ spectra were recorded in the 310-1000 nm wavelength range at 50°, 60° and 70° angles of incidence. To build an appropriate model in the case of sensitive thin films, it is necessary to know the optical data of the substrate material, which can be determined from measurements performed on the bare substrate. Measured data was evaluated by the Cauchy dispersion relation extended with the Urbach-absorption (Ferlauto *et al.* 2002). Ellipsometric measurements performed on the sensors made from α -Fe₂O₃ decorated with MWCNTs can be evaluated by a three phase model: known substrate, sensitive layer and surface roughness. Increasing absorption of the sensitive layer towards the UV region was observed in both cases. This kind of absorption was modeled by the Tauc-Lorentz dispersion formula (Jellison Jr. and Modine 1996). The results of optical data from ellipsometric measurements are presented in Fig. 3.

It is determined from these dates that the thickness of the sensitive thin films made from α -Fe₂O₃ decorated with MWCNTs was equal \sim 36 nm.

3. Results and discussion

A response of the prepared sensors made from α -Fe₂O₃ decorated with MWCNTs (25:1) to the influence of ethanol vapors (C₂H₅OH) was measured using a special system described in (Adamyany *et al.* 2007, Aroutiounian *et al.* 2013a). Samples were placed in hermetic chamber. The certain quantity of liquid spirit is placed in the chamber for corresponding concentration of alcohol vapors supply. The sensor is put on the heater which allows raising temperature of the sensor working body up to 350°C. The sensor resistance changing depending on the working body temperature and alcohol vapors concentration was measured using the special computer program. The management program is written by Dr. A. Adamyany in the Borland Delphi 6.0 environment. All measurements were carried out at the sensor applied voltage 0.5 V.

The investigations of ethanol sensors manufactured by us from α -Fe₂O₃/MWCNTs (25:1) nanostructure were carried out. The response to ethanol vapors was measured for these sensors. The sensor response was determined as ratio $R_{\text{air}}/R_{\text{gas}}$, where R_{air} is the sensor resistance in air and R_{gas} is the sensor resistance in the presence of alcohol vapors in air. Note that the response time is determined as the time required for a sensor to reach 90% of its maximum response. The recovery time is determined as the time taken by the sensor to get to 10% of the value of its maximum resistance.

The results of measurements of the response to ethanol vapors at different temperature of the sensor work body are presented on Fig. 4. In these measurements the ethanol vapors concentration was equal to 5000 ppm. The sensor resistance is decreased by 2 orders of magnitude under the influence of 5000 ppm alcohol vapors at the working body temperature 200°C. The response more than 300 was detected at the working body temperature 300°C. These results are much better in comparison results obtained for sensors made on nanostructured Fe₂O₃ (Pawar *et al.* 2012). The response and recovery times for the sensor made from α -Fe₂O₃/MWCNTs (25:1) were found to be 2 min and 8 s at 300°C. The large values of the response time is related by that for obtaining of the

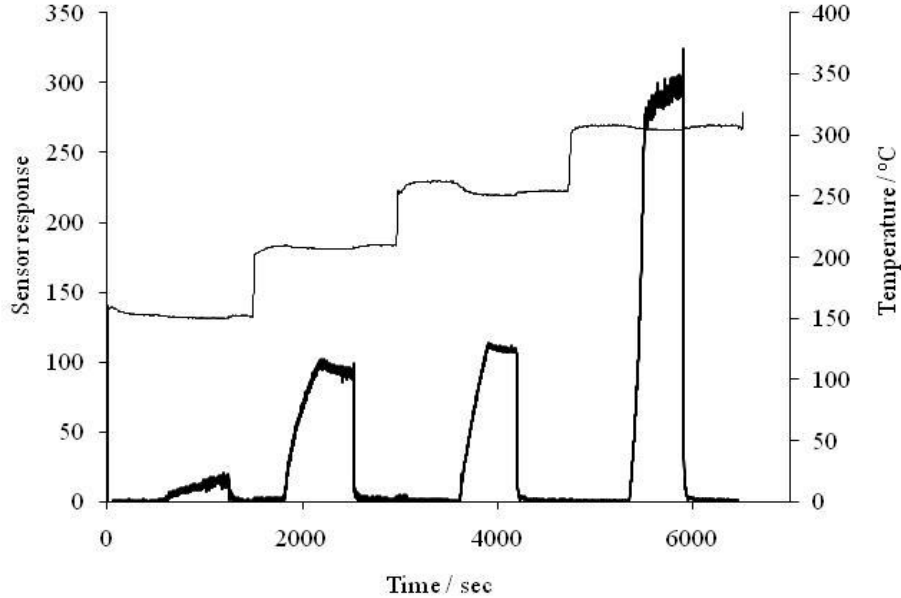
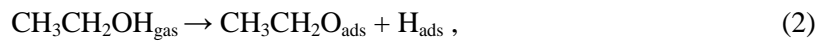


Fig. 4 Response ($R_{\text{air}}/R_{\text{gas}}$) to ethanol vapors for sensors made from $\alpha\text{-Fe}_2\text{O}_3$ decorated with MWCNTs at different temperatures of the work body. The concentration of ethanol vapors was 5000 ppm. Data about the work body temperature is given on Fig. 4 on the right y-axis and response ($R_{\text{air}}/R_{\text{gas}}$) – on the left y-axis

appropriate concentration of ethanol vapors, as it has already been mentioned, the stringently certain quantity of liquid spirit is putted in the chamber. The sensor response time, counted from the moment of putting of liquid alcohol in the chamber till the moment of achievement of 90% of the maximum response, includes a time required the full evaporation of alcohol.

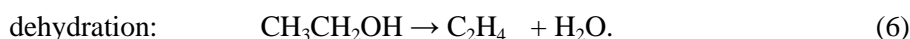
It is known that the sensitivity of resistive gas sensors is caused by chemical reactions taking place between the target gas molecules and the ions adsorbed on the surface of the sensing material. In particular, the following transformation may be take place on MWCNTs surface:



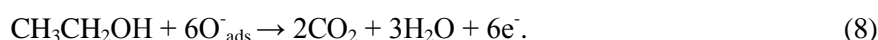
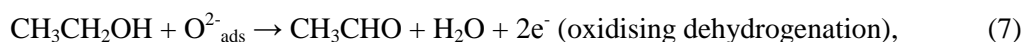
As one can see, the ethanol in the gas phase is transforming into ethoxide ($\text{CH}_3\text{CH}_2\text{O}$) and hydrogen atom adsorbed on the carbon nanotubes surface. Then, ethoxide may be transformed to acetaldehyde (CH_3CHO) and adsorbed hydrogen. Further adsorbed hydrogen atoms lose their electrons, transferring them to the crystal lattice. As a result, the structure resistance is decreased. However, it should be noted that due to the fact that the concentration of the free charge carriers in MWCNTs is very large, generated according to the Eq. (4) charge carriers do not make the essential contribution to conductivity, i.e., do not provide appreciable change of the sensor resistance.

The ethanol detection on the $\alpha\text{-Fe}_2\text{O}_3/\text{MWCNTs}$ (25:1) surface is the consequence of two

processes: interaction of alcohol vapors with both carbon nanotubes (reactions 2-4) and semiconductor metal oxide surface. The transformation of the ethanol can take place by two ways on the metal oxide surface (Ivanovskaya *et al.* 2003)

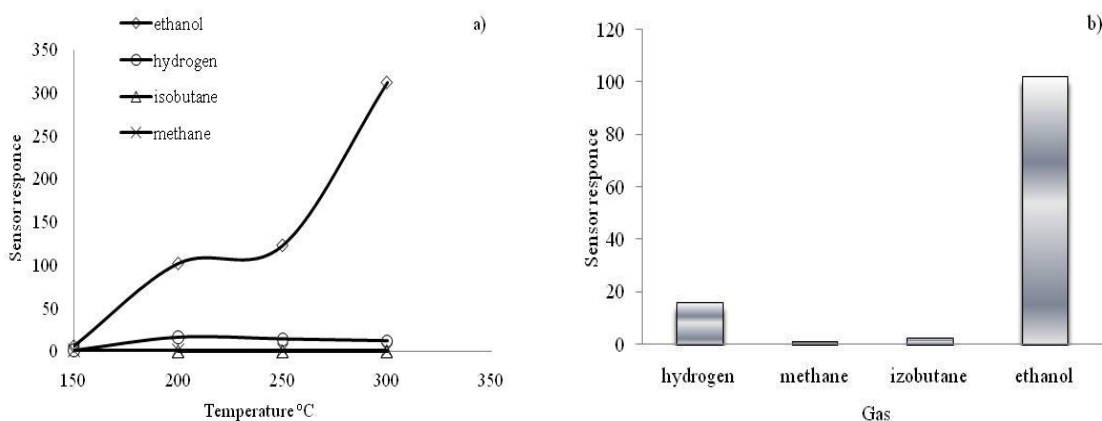


Further, the components originated from the dissociation components interact with the oxygen ions adsorbed on the oxide surface. That leads to the conductivity change. The following reactions, for example, may be taking place on the metal oxide surface



Probably, decoration of $\alpha\text{-Fe}_2\text{O}_3$ with MWCNTs provides existence a depletion space charge layer owing to the adsorption of oxygen from air. The influence of electrons generated by ethanol vapors as a result of simultaneously proceeding reactions (2-8) leads to sharp decrease in the sensor resistance. Thus, the metal oxide presence provides significant response of sensors made from MWCNTs.

Fig. 5(a) represents the results of measurements of the response of investigated sensors made from $\alpha\text{-Fe}_2\text{O}_3/\text{MWCNTs}$ (25:1) to various gases at different temperatures of the work body. As shown, the tested sensors are not sensitive to methane, but some response to hydrogen and isobutane is noticed at temperatures above 200°C. It shows also the selectivity of prepared sensor to ethanol is much higher as compared to other gases (Fig. 5(b)). In spite of the fact that prepared sensor showed maximum response to ethanol vapors at 300°C, but its response considerably exceeds response to other gases already at temperature 200°C. Note that response to ethanol of our sensors from $\alpha\text{-Fe}_2\text{O}_3/\text{MWCNTs}$ (25:1) at 300°C is remarkable higher than the response of non-decorated sensor (Pawar *et al.* 2012) at 350°C.



(a) Response ($R_{\text{air}}/R_{\text{gas}}$) to all tested gases for sensors made from $\alpha\text{-Fe}_2\text{O}_3$ decorated with MWCNTs at different temperatures of the work body. The gas concentration was 5000 ppm

(b) Selectivity of sensors made from the $\alpha\text{-Fe}_2\text{O}_3/\text{MWCNTs}$ (25:1) nanostructure at 200°C

Fig. 5 Response to all tested gases and selectivity for sensors made from $\alpha\text{-Fe}_2\text{O}_3$ decorated with MWCNTs

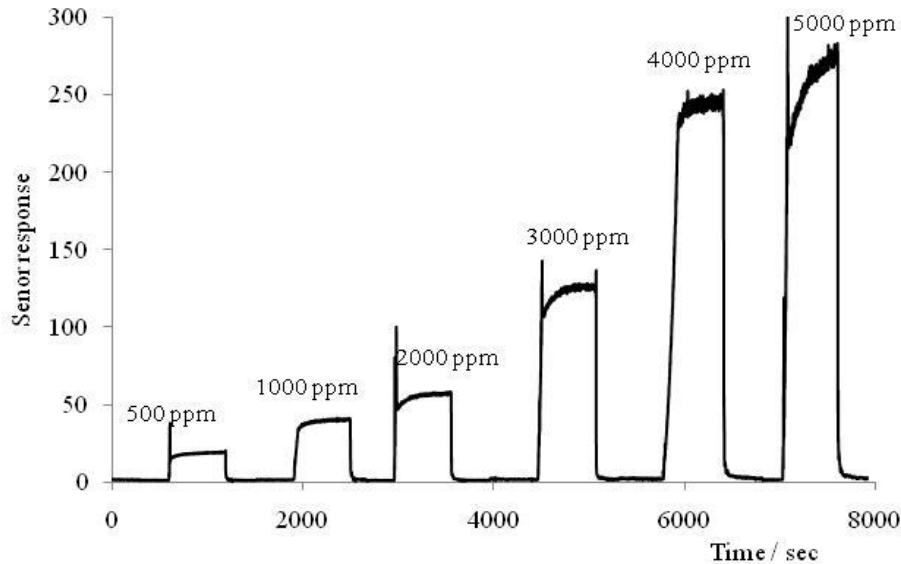


Fig. 6 Response of sensors made from the $\alpha\text{-Fe}_2\text{O}_3/\text{MWCNTs}$ (25:1) nanostructure to different concentrations of ethanol vapors. Temperature of the sensor work body was equal 300°C

The results of the response measurements of prepared by us sensors made from the $\alpha\text{-Fe}_2\text{O}_3/\text{MWCNTs}$ (25:1) nanostructure under influence of different concentration of ethanol vapors are presented in Fig. 6. The work body temperature was 300°C .

This structure is sensitive to ethanol vapors since its concentration 500 ppm. Monotonous growth of sensor response with the increase in alcohol vapors concentration allows not only detecting small quantities of ethanol vapors in air, but also determining its concentration with high accuracy.

4. Conclusions

Thin film sensors made from $\alpha\text{-Fe}_2\text{O}_3$ decorated with MWCNTs were manufactured and investigated. The sensor response was measured at different temperatures of the sensor work body and at different concentration of ethanol vapors.

- It was established that prepared sensors showed appreciable response to ethanol vapors already at temperature of the work body 150°C .
- Prepared sensors showed maximum response (312) to ethanol vapors at the work body temperature 300°C .
- The response to other tested gases (hydrogen, methane, isobutane) is lower as compared to response to ethanol.
- Sensors made from the $\alpha\text{-Fe}_2\text{O}_3/\text{MWCNTs}$ (25:1) nanostructure can be used for detection of low concentration of ethanol vapors (since the concentration 500 ppm) and also for determination of their concentration.

Acknowledgments

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