Influence of chemical composition and structural factors of Fe_2O_3/In_2O_3 sensors on their selectivity and sensitivity to ethanol☆

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Abstract

The influence of structure, phase composition, system dispersity and particle morphology of Fe_2O_3- and In_2O_3-based thin film layers on the electrical parameters of the corresponding gas sensors was studied in this paper. High selectivity and sensitivity of heterostructural γ-Fe_2O_3/In_2O_3 thin film sensors in the detection of alcohols has been here established. As it was found, the above mentioned sensors are almost insensitive to NO_2, O_3, CO and CH_4 at the operating temperature which is optimal for alcohol detection (300 °C). The peculiarities of sensor behaviour are discussed taking into account the activity of Fe_2O_3 layers in the reductive-oxidative and acid–base processes accompanying the adsorption of CH_3OH and C_2H_5OH molecules at the oxide surface.

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1. Introduction

Iron oxides are known to be widely used materials as active element of ceramic and thick film gas sensors, but there are only few items of information concerning the application of iron oxide as thin film sensing layers. This paper is mainly aimed at the determination of the influence of some structural features of Fe_2O_3–In_2O_3 films on their gas-sensitive properties. Great attention was paid to heterojunction structure Fe_2O_3–In_2O_3/In_2O_3, which is found to be advanced material for reducing gas detection.

From the available references, it follows that the sensitivity of ceramic sensors based on Fe_2O_3 towards reducing gases is rather low [1–3]. However, doping of Fe_2O_3 with quadrivalent metal ions (Sn, Ti, Zr), as well as modifying of a sensitive material with SO_4^{2-} ions, can significantly enhance the gas-sensitive properties of the corresponding sensors regarding ethanol and hydrocarbons [3], addition of Fe_2O_3 to SnO_2 leads to an improved response of thick films towards ethanol [4]. There are also some items of literature concerning the effect of Fe_2O_3 additives on the properties of In_2O_3 based sensors. MoO_3–SnO_2 sensors are known to be extremely selective regarding ethanol [5] and may serve as reference point when detection of alcohol is considered. It is known that the doping of SnO_2 and In_2O_3 with γ-Fe_2O_3 increases their sensitivity to oxidising gases (O_3, NO_2) [6]. But there are only little references regarding γ-Fe_2O_3 using as thin film sensors towards reducing gases. And the data available are not sufficient in order to realize full picture of iron-containing material application in gas sensor industry.

2. Experimental

Gas-sensitive properties of thin film sensors containing one oxide layer (Fe_2O_3, In_2O_3) and bi-layers (Fe_2O_3/In_2O_3, Fe_2O_3–In_2O_3/In_2O_3) have been investigated here. Sensor response values to CH_3OH, C_2H_5OH (100–500 ppm), CH_4, CO (50 ppm) were estimated.

The sensitive elements were formed from the stabilised sols of the corresponding metal hydroxides that were obtained by sol–gel technique. The procedure of sol preparation used in this study consisted in the following steps: (i) forced hydrolysis of inorganic metal salt solution ($FeCl_2$, $Fe(NO_3)_3$, $In(NO_3)_3$) with base agent (NH_3); (ii) precipitation of metal hydroxide followed by its separation; (iii) formation of sol through peptization of the deposit with peptizing agent or as a result of self-peptization. Methods of $Fe_2O_3 \cdot nH_2O$ sol synthesis providing the formation of both

γ-Fe$_2$O$_3$ and α-Fe$_2$O$_3$ oxides under heating were used; besides, two types of γ-Fe$_2$O$_3$ layers (prepared via Fe(OH)$_2$ as an intermediate (I-γ-Fe$_2$O$_3$) and via Fe$_3$O$_4$ (II-γ-Fe$_2$O$_3$) sols) were here studied. The composites were prepared by both combined Fe(OH)$_2$ and In(OH)$_3$ hydroxide precipitation followed by their oxidation with oxygen (a flow of air was passed through the suspension during 5–6 h at 30 °C) and by mixing of individual sols of Fe$_2$O$_3$ and In(OH)$_3$ in required proportions.

To form thin film layers, sols were deposited onto polycrystalline Al$_2$O$_3$ substrates (3 mm × 3 mm × 0.25 mm) supplied with Pt interdigital electrode structure on the front side and Pt meander heater on the backside. An In$_2$O$_3$ sub-layer was preliminary deposited onto the substrate in order to knowingly form heterojunction structure and provide suitable sensor electroconductivity. For comparison, single-layer sensors, consisting of only In$_2$O$_3$ or Fe$_2$O$_3$ have also been studied. The samples were dried at 25 °C followed by annealing at 300 °C in air. The sensor response was calculated as $S = \Delta G / G_{air}$, where G and G_{air} is the conductivity of sensitive layer in alcohol–synthetic air mixture and in synthetic air consequently (RH 30%).

The structure of the simple oxides (Fe$_2$O$_3$, In$_2$O$_3$) and nanocomposites (Fe$_2$O$_3$–In$_2$O$_3$) has been characterised by means of X-ray diffraction (HZG-4A diffractometer, Co Kα radiation, Ni-filter, angle range 20–70°).

3. Results and discussion

The variation of electrical conductivity, system dispersity and oxide particle sintering of both simple oxides (Fe$_2$O$_3$, In$_2$O$_3$) and Fe$_2$O$_3$–In$_2$O$_3$ composites as a function of the synthesis conditions, components ratio and operating temperature was analysed in the paper.

Fig. 1 clearly illustrates the fact that the sensors based on single Fe$_2$O$_3$ and In$_2$O$_3$ layers have practically the same
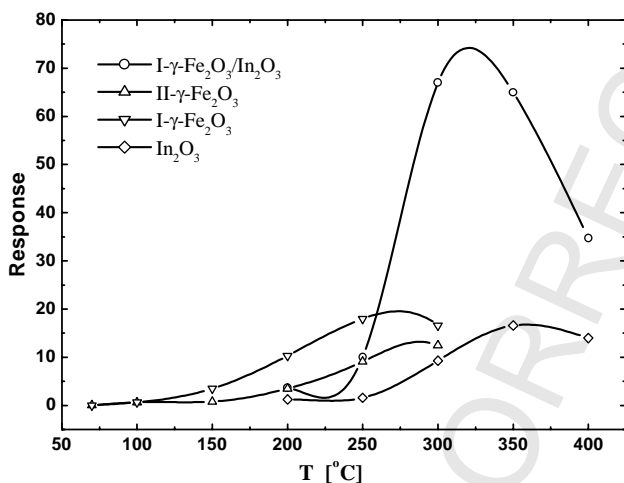

Fig. 1. Temperature-dependent response of In$_2$O$_3$ and Fe$_2$O$_3$ thin film sensors to C$_2$H$_5$OH (100 ppm, RH 30%).
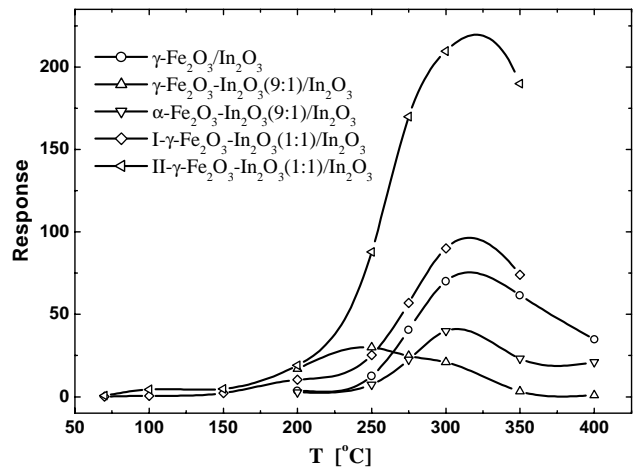
Fig. 2. Temperature-dependent response of In$_2$O$_3$–Fe$_2$O$_3$ bi-layer thin film sensors to C$_2$H$_5$OH (100 ppm, RH 30%).

response values to ethanol, but they slightly differ regarding the optimal operating temperature. The sensors based on γ-Fe$_2$O$_3$/In$_2$O$_3$ heterostructure (bi-layer sensors) are characterised by significantly higher sensitivity to ethanol than the single-layer (γ-Fe$_2$O$_3$, In$_2$O$_3$) ones. In this case, In$_2$O$_3$ sub-layer not only provides the sufficient γ-Fe$_2$O$_3$/In$_2$O$_3$ film conductivity, but it has a clear influence on the sensor sensitivity. We suppose that the important point influencing strongly the activity of sensor is the presence of phase interface in such a sample. Note, that both In$_2$O$_3$ and Fe$_2$O$_3$ layers demonstrates typical semiconducting behaviour [7].

The addition of In$_2$O$_3$ (10 mol%) to γ-Fe$_2$O$_3$ layer leads to decreasing of the γ-Fe$_2$O$_3$/In$_2$O$_3$ sensor response to ethanol (Fig. 2). According to the XRD data, γ-Fe$_2$O$_3$–In$_2$O$_3$ (9:1) composite has the structure of In^{3+} in γ-Fe$_2$O$_3$ lattice solid solution (Fig. 3); certain amount of C–In$_2$O$_3$ phase is also presented. On the contrary, further increasing of In$_2$O$_3$ content within γ-Fe$_2$O$_3$ layer of γ-Fe$_2$O$_3$/In$_2$O$_3$ structure up to 50 mol% causes abrupt rising of its sensitivity, which even overwhelms the sensitivity of γ-Fe$_2$O$_3$/In$_2$O$_3$ sample. In comparison with γ-Fe$_2$O$_3$–In$_2$O$_3$ (9:1) sample, γ-Fe$_2$O$_3$–In$_2$O$_3$ (1:1) one probably consists of two highly dispersive phases (see Fig. 3) with great phase interface. From the XRD data of the sample it is difficult to determine exactly phase composition of the sample because of the similarity of γ-Fe$_2$O$_3$ and C–In$_2$O$_3$ crystal lattices. The growth of γ-Fe$_2$O$_3$–In$_2$O$_3$ (1:1) dense layer is due to the formation of numerous bonds between the oxide particles within this composite. Both developed surface of contacting oxides and their specific morphology results in significant improvement of γ-Fe$_2$O$_3$–In$_2$O$_3$ sensitive species.

Moreover, Fe$_2$O$_3$–In$_2$O$_3$/In$_2$O$_3$ sensor sensitivity hardly depends on the crystal structure of the active layers, in particular, Fe$_2$O$_3$ phase. There are two possible Fe$_2$O$_3$ phases, suitable under operating temperatures used γ-Fe$_2$O$_3$ and α-Fe$_2$O$_3$. In the case of the pure γ-Fe$_2$O$_3$, the response achieves its maximum value at lower temperatures (250 °C)
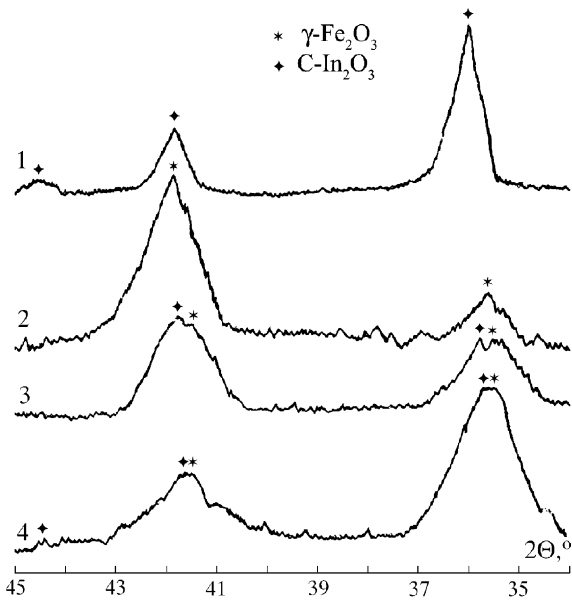
Fig. 3. XRD patterns of In_2O_3, γ-Fe_2O_3 and γ-Fe_2O_3–In_2O_3 composites annealed at 300 °C (1 h): (1) In_2O_3; (2) γ-Fe_2O_3; (3) γ-Fe_2O_3–In_2O_3 (9:1); (4) γ-In_2O_3–Fe_2O_3 (1:1).

than in the case of α-Fe_2O_3 (300 °C). Higher activity of γ-Fe_2O_3 phase is associated with specificity of its structure: the presence of metal cation vacancies within crystalline lattice and the readiness of $Fe^{2+} \leftrightarrow Fe^{3+}$ transformation under the exposure of gas media. Besides, partial reversible reduction of γ-Fe_2O_3 layer to form α-Fe_2O_3 is possible. The product of reduction is highly dispersive and strongly reactive and resembles the behaviour of γ-phase of Fe_2O_3. When methanol detection is considered, one can observe the same regularities inherited to ethanol, excepting the lower response (Fig. 4).

The conditions of γ-Fe_2O_3 sol preparation influence also the γ-Fe_2O_3 phase microstructure and the sensor activity (Table 1).
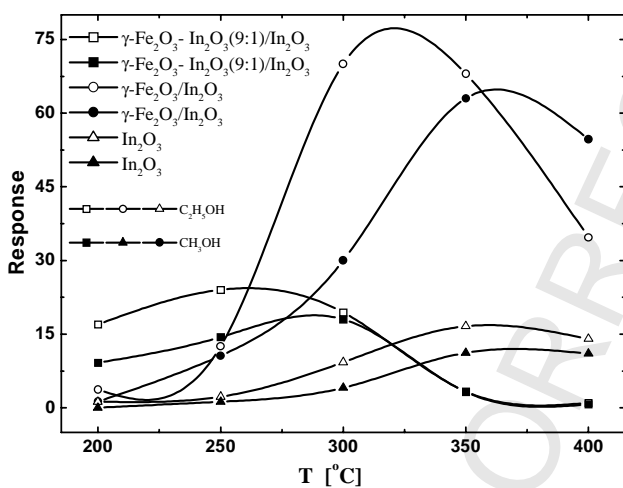

Fig. 4. Comparison of In_2O_3 and Fe_2O_3 based thin film sensor behaviour in C_2H_5OH and CH_3OH media (100 ppm, RH 30%).

Table 1
The response of thin film sensors of various compositions to ethanol and CO

Sensor	dG/G CO (50 ppm)	T (°C)	dG/G EtOH (100 ppm)	T (°C)
In_2O_3	0.45	350	15	350
α-Fe_2O_3–In_2O_3 (9:1)/In_2O_3	0.9	300	45	300
I-γ-Fe_2O_3/In_2O_3	1.25	350	65	300
I-γ-Fe_2O_3–In_2O_3 (9:1)/In_2O_3	0.9	250	25	250
I-γ-Fe_2O_3–In_2O_3 (1:1)/In_2O_3	0.8	250	125	300
II-γ-Fe_2O_3–In_2O_3 (1:1)/In_2O_3	0.1	350	200	300
I-γ-Fe_2O_3	−0.9	150	12	250
II-γ-Fe_2O_3	0.1	250	17	300

The layers having γ-Fe_2O_3 crystal structure show different particle sizes; it depends on the method of sol synthesis. γ-Fe_2O_3 films prepared by oxidation of $Fe(OH)_2$ with molecular oxygen (method I) are highly dispersive ($\sim$5 nm), while II-γ-Fe_2O_3 sample synthesised by oxidation of Fe_3O_4 sol with oxygen is consisted of spherical particles of greater dimension ($\sim$20 nm) and of less developed phase interface. As a result, sensor based on simple I-γ-Fe_2O_3 demonstrates higher response than II-γ-Fe_2O_3 one (see Fig. 1). But in the case of γ-Fe_2O_3–In_2O_3 composites, prepared by γ-Fe_2O_3 and $In(OH)_3$ sol mixing this regularity has reverse direction (see Fig. 2). The explanation follows from the comparison of the two simple oxide dispersity: the less grain size of an oxide the more homogeneous system one can get under sol mixing followed by the obtained powder thermal treatment. Thus, greater dimension of II-γ-Fe_2O_3 phase particles favours the formation of less homogeneous and, consequently, more active layers than in case of I-γ-Fe_2O_3.

Both γ-Fe_2O_3/In_2O_3 and γ-Fe_2O_3–In_2O_3/In_2O_3 sensors were found to be selective regarding alcohol vapours. These sensors are practically insensitive to CO, CH_4, NO_2 and O_3 at the temperature which is optimal for ethanol detection (300 °C). Table 2 shows that both γ-Fe_2O_3/In_2O_3 and γ-Fe_2O_3–In_2O_3 (1:1)/In_2O_3 thin film sensors are essentially more sensitive than SnO_2–MoO_3 samples which demonstrate high sensitivity to ethanol.

Dynamical parameters of the studied sensors are found to be suitable for thin film layers. Both response and recovery times lay in the range of 50–60 s at optimal operating temperatures (100 ppm C_2H_5OH, 300–350 °C, RH 30%).

As it was noted earlier, the systems consisting of two contacting phases, In_2O_3 and γ-Fe_2O_3 are extremely sensitive to ethanol. In the case of γ-Fe_2O_3–In_2O_3 (1:1) com-

Table 2
The response of thin film sensors based on various oxides to ethanol (500 ppm) comparison

| Sensor	dG/G	T (°C)
In_2O_3	15	350
γ-Fe_2O_3/In_2O_3	165	300
SnO_2	30	400
SnO_2–MoO_3	80	300

posite, grain size is less and surface of phase interface is greater (and, consequently, response values are greater) than those, typical for γ-Fe_2O_3/In_2O_3 heterocontact. The presence of two phases within composite favours to the effective course of multi-step process of both reductive–oxidative and acid–base interactions. Thus, alcohol detection is considered as a process involving the two interactions between the detected molecules and the particles of the oxide element [2]. The obtained results concerning Fe_2O_3–In_2O_3 and SnO_2–MoO_3 based sensor were here interpreted assuming a different activity of the above systems in both processes. The possibility of intermediate further oxidation, the second phase take part in, is considerably increases sensor response values to ethanol.

Two ways of alcohol (for example, ethanol) molecule conversion are possible at oxide surface, dehydrogenation (1) and dehydration (2) [8–10].

$$CH_3CH_2OH \rightarrow CH_3CHO + \tfrac{1}{2}H_2 \quad \text{(dehydrogenation)} \quad (1)$$

$$CH_3CH_2OH \rightarrow C_2H_4 + H_2O \quad \text{(dehydration)} \quad (2)$$
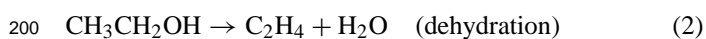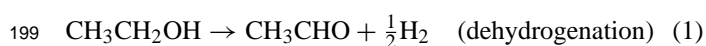
Besides, further oxidation of the formed products (foremost, oxidation of H atoms) is possible at oxide surface; it should result in sensor response growth. Thus, the process (1) most often proceeds through the oxidising dehydrogenation mechanism

$$CH_3CH_2OH \overset{O^{2-},O^-}{\rightarrow} CH_3CHO + H_2O$$

$$\text{(oxidising dehydrogenation)} \quad (3)$$
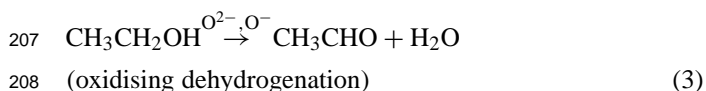
As we established earlier [11], in the indicated process lattice oxygen is only participating at high operating temperatures (300–400 °C). The role of adsorbed oxygen consists in regenerating of partially reduced oxide surface. The oxidising dehydrogenation of alcohol (3) is heterolytic catalytic reaction. The process involves both reductive–oxidative and acid–base steps [12]. In particular, alcohol molecule adsorption at metal cation, which plays role of Lewis centre, is related to acid–base reaction. The relative measure of oxide activity in the oxidation reactions can be oxygen-oxide surface bonding energy. In fact, the less the energy of oxygen atom isolation from an oxide surface the higher the oxide oxidising ability. The reactivity of oxides in acid–base reactions depends on the electronegativity of cations M^{n+}.

$$\chi = \chi_0(2n + 1) \quad (4)$$

where χ_0 is the Pauling's electronegativity and n the ion charge. The electronegativity can be used as the measure of Lewis acid site activity. The adsorption of alcohol molecules at Lewis sites is going with great output. Complete oxidation of intermediate products is possible at the surface of the oxide, which is characterised by small values of M–O binding energy and electronegativity. According to the electronegativity (χ) increasing the studied oxides can be placed as follows: $In_2O_3 < Fe_2O_3 < SnO_2 < MoO_3$ (Table 3) [13].

Table 3
The metal–oxygen binding energy for some oxides

Metal oxide	E_{M-O} (kcal/g per atom)	Electronegativity
MoO_3	91	30.55
SnO_2	70	17.64
Fe_2O_3	56	13.72
In_2O_3	–	12.46

But in the general case, reductive–oxidative and acid–base properties of oxide surface may not correlate.

Thus, detection of ethanol using oxide materials, where metal is greatly electronegative (MoO_3) is not accompanying by complete oxidation of intermediates, but is characterised by striking selectivity. In the range of considerable M–O bonding energy values, donor–acceptor interaction between alcohol molecules and $M^{m+}O^{2-}$ species become clearly apparent. Oxides, containing Mo(VI) cations, which are strong Lewis acids, are very active. In fact, the low bonding energy and strongly basic properties of an oxide promote further oxidation of intermediates of alcohol molecule transformation (Fig. 5), thereby, increasing the corresponding sensor response values.

The oxides, which are characterised by the possibility of metal ion reduction without oxide phase state modification, have the greatest ability to promote oxidising dehydrogenation processes. For instance, such oxides as MoO_3, In_2O_3 and Fe_2O_3 are inclined to facile the changing of metal ion oxidising state: Mo(VI) $\leftrightarrow$ Mo(V), In(III) $\leftrightarrow$ In(II), Fe(III) $\leftrightarrow$ Fe(II), while oxide phase remains original.

The sensors based on heterojunction oxide show considerable response to alcohol (ethanol, methanol) vapours. The heterojunction between an oxide and solid solution phases appears to be very active in both adsorption and oxidation of alcohol. The presence of two types of centres possessing different reductive–oxidative and acid–base properties, and participating in the processes of the alcohol molecule transformation is essential requirement to achieve high sensor response when alcohol detection is considered. The centres of one type can suitably participate in adsorption–desorption processes of alcohol molecules, whereas complete oxidation of intermediates is effectively going at the centres of another type.
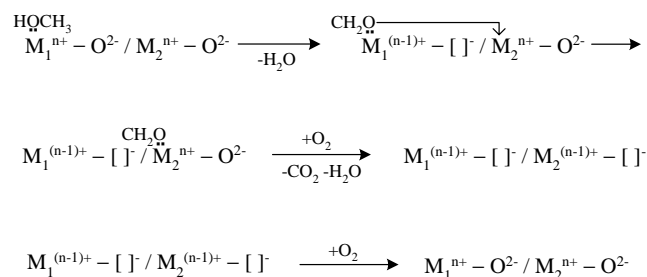

Fig. 5. The scheme of alcohol oxidation process at oxide phase interface by the example of methanol (CH_3OH) molecule oxidation.

The experimental data regarding both catalytic and gas-sensitive properties of most of semiconducting oxides are not in accord with prevailing conception, asserting that the alcohol detection is predominantly realised as dehydration process (2), when oxide with acidic properties are used, and as dehydrogenation (1) at base oxide surface [9]. Thus, MoO_3 possesses strong acidic behaviour. At the same time, it is one of the most efficient catalysts of methanol conversion into formaldehyde (reaction of dehydrogenation) [14]. By reason of MoO_3 high catalytic activity, it being added to SnO_2 species evokes the increasing the corresponding sensor sensitivity towards alcohols [10]. However, in this case, after-oxidation of formaldehyde, generated during dehydrogenation step, to form CO_2 and H_2O is impossible because of great Mo–O bond strength within MoO_3. Using this fact one can explain lesser response of SnO_2–MoO_3 sensors to ethanol in comparison with Fe_2O_3–In_2O_3 ones.

The analysis of the literature available regarding catalytic alcohol decomposition allows us to conclude that the prevailing in gas sensor industry oxides are more active in dehydrogenation reactions rather than in dehydration. Relying on numerous experimental data, in [12] have been calculated the values of catalytic reactivity ($\ln K$) for different oxides in both dehydrogenation and dehydration of isopropyl alcohol at the same conditions of catalysis. Table 4 represents the values of catalytic reactivity for the oxides used in this paper.

It is important to note, the regularities obtained for isopropyl alcohol are proper for ethanol as well. However, there are no valid experimental data in the literature, allowing us to realise the detailed calculation of catalytic properties of the oxides regarding ethanol decomposition.

Mass-spectrometry analysis of the desorbed products of ethanol transformation at SnO_2 confirms the course of alcohol decomposition through predominantly the dehydrogenation in a wide range of temperatures (50–400 °C). Thus, the concentration of acetaldehyde (CH_3CHO) in the desorbed products exceeds ethylene (C_2H_4) content more than 10 times.

From the evidence reported one can conclude that the main process determining the efficiency of alcohol detection using semiconducting oxides is oxidising dehydrogenation steps; dehydration have minor significance only. This point should be taken in consideration when selecting advanced gas-sensitive composites for efficient alcohol detection.

Table 4
The comparison of oxide catalytic ability in isopropyl alcohol decomposition process

Oxide	Reactivity ($\ln K$)	
	Dehydrogenation	Dehydration
MoO_3	1.91	0.50
In_2O_3	1.54	0.82
Fe_2O_3	1.52	0.72
SnO_2	1.01	0.44

4. Conclusions

Fe_2O_3–In_2O_3 based thin films are found to be selective and highly sensitive to alcohol vapour at moderate (300 °C) operating temperature. It was found that the composites, consisted of two contacting phases (In_2O_3 and Fe_2O_3) with large phase interface are preferable materials for gas sensing. In particular, γ-Fe_2O_3–In_2O_3 (1:1)/In_2O_3 sensor has the highest dispersity and the most developed phase interface among the Fe_2O_3–In_2O_3 composites and it is characterised by the best functional parameters. The most sensitive materials consist of two layers. In this case, the specific hetero-junction is forming providing the differentiation between receptor and transducer functions, that improves sensor performance.

It has also been established that the detection of alcohols by using active layers based on investigated oxides is mainly going on the way of oxidising dehydrogenation.

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Biographies

Maria Ivanovskaya received her degree in chemistry in 1980 from Belarus State University in the field of photochemistry. Since 1989 she has been worked in Scientific and Research Institute of Physical and Chemical Problems (Belarus State University). Her main scientific interest is solid state chemistry in applications to catalysis and semiconductor gas sensors, structural features of nanosized oxides (SnO_2, MoO_3, In_2O_3, Fe_2O_3) and oxide composites. She is co-author of more than 200 papers on the indicated topics.

Dzmitry Kotsikau graduated from the Belarus State University in 2001 with honours, in the same year entered the post-graduate courses. Now he is working in the field of solid state chemistry and semiconductor gas sensors. His main scientific interests are Fe_2O_3–In_2O_3 nanosized composites, their structural and gas-sensitive characterization.

Guido Faglia received an MSc degree from the Polytechnic of Milan in 1991 with a thesis on gas sensors. Since then he has been studying to obtain the PhD in electronics by the University of Brescia. In 1992 he has been appointed as a Researcher by the Thin Film Lab at the University of Brescia. He is involved in the study of the interactions between gases and the tin oxide surface and in sensor electrical characterization. In 1996 he has received the PhD degree by discussing a thesis on semiconductor gas sensors.

Paolo Nelli received his MSc degree in physics from the University of Pavia in 1987, after then he joined the Gas Sensor Lab at the University of Brescia, where at present he carries out his research activity on gas sensors. He held a permanent position at the Department of Chemistry and Physics for Materials of the University of Brescia, his interests are focused on the preparation and characterisation of gas sensors based on semiconducting metal oxides. He is co-author of about 40 papers on gas sensors and material science and 30 presentations at international congresses on the same topics.