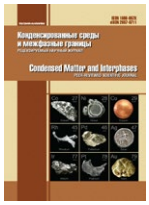

ISSN 1606-867X (Print)
ISSN 2687-0711 (Online)
Condensed Matter and Interphases
Kondensirovannye Sredy i Mezhfaznye Granitsy
https://journals.vsu.ru/kcmf/

Original articles

Research article
https://doi.org/10.17308/kcmf.2025.27/13328

Biotemplate synthesis of In_2O_3-Pd for room temperature sensor of hydrogen

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Abstract

Objective: The solution to the urgent task of creating compact gas analyzers capable of long-term autonomous operation in hard-to-reach places is related to the development of sensors with reduced energy consumption. The aim of this work was to create a room temperature hydrogen sensor, as it is the sensor's heating that contributes significantly to the energy consumption of the entire device.

Experimental: To solve this problem, a new method for the synthesis of a nanomaterial based on In_2O_3 with a 3 % palladium additive was developed, which differs significantly from the common methods of sol-gel synthesis and hydrothermal synthesis. This was due to the fact that at low sensor temperatures, minimizing the effect of humidity is crucial. Performing the synthesis in an aqueous environment leads to the formation of a large number of hydroxyl groups on the surface, which attract water. In our work, the nanomaterial was prepared by sintering a cellulose fiber pre-impregnated with a solution of indium nitrate (+3) and tetraammine palladium nitrate (+2). According to X-ray phase analysis, the powder sintered at a temperature of 500 °C consists mainly of the triclinic phase of indium oxide (+3). According to scanning electron microscopy, the samples largely retained the reproducible characteristic macrostructure of the cellulose template. The electrophysical characteristics of the nanomaterial obtained at room temperature showed the possibility of determining hydrogen in the air. The detection limit is less than 10 ppm.

Conclusions: The sensitivity of our hydrogen sensor at room temperature is higher than that of sensors described in other publications. The effect of humidity on sensor readings is minimized.

Keywords: Metal oxide sensors, Biotemplated synthesis, Hydrogen, Room temperature, Indium oxide, Palladium

Funding: RSV and TSYu acknowledge the grant of the Ministry of Science and Higher Education of the Russian Federation (Project FZGU-2023-0006) in part of material characterization.

Acknowledgements: The SEM studies and X-ray phase analysis were performed on the equipment of the Center for the Collective Use of Scientific Equipment of Voronezh State University.

For citation: Shaposhnik A. V., Arefieva O. A., Zviagin A. A., Brezhnev N. Ya., Vysotskaya E. A., Vasiliev A. A., Ryabtsev S. V., Turishchev S. Ya. Biotemplate synthesis of In_2O_3-Pd for room temperature sensor of hydrogen. *Condensed Matter and Interphases*. 2025;27(4): 689–695. https://doi.org/10.17308/kcmf.2025.27/13328

Для цитирования: Шапошник А. В., Арефьева О. А., Звягин А. А., Брежнев Н. Ю., Высоцкая Е. А., Васильев А. А., Рябцев С. В., Турищев С. Ю. Биотемплатный синтез In_2O_3-Pd для низкотемпературного сенсора водорода. *Конденсированные среды и межфазные границы*. 2025;27(4): 000-000. https://doi.org/10.17308/kcmf.2025.27/13328

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1. Introduction

The determination of hydrogen and other combustible explosive gases in hard-to-reach places is an important practical task. Gas sensors are commonly used to solve this problem, including thermocatalytic, electrochemical, and semiconductor sensors. Thermocatalytic sensors are simple and inexpensive, but they have low sensitivity and selectivity. Electrochemical sensors, which are similar to fuel cells, have high sensitivity and selectivity, but they have a limited operational duration due to the consumption of the reactant. High sensitivity and high selectivity can be achieved using semiconductor sensors, but their application usually requires maintaining an operating temperature of around 300 °C. The significant energy consumption makes it difficult to create autonomous gas analyzers based on semiconductor sensors that can operate in remote areas for extended periods.

There are several approaches to solving the problem of reducing energy consumption by semiconductor sensors. One approach is to miniaturize the sensor and use special dielectric substrates with low heat capacity [1]. Another approach is pulse heating, which not only reduces energy consumption but also increases the sensitivity and selectivity of the analysis in some cases [2]. In recent years, there have been many publications that propose a third approach to solving this problem: the synthesis of gas-sensitive materials that can perform gas analysis without heating the sensor. In English literature, this approach is known as a "room temperature sensor." These sensors can be used to detect various gases in the air, including both reducing gases such as ammonia [3–5] and hydrogen sulfide [6-8], and oxidizing gases such as NO_2 [9–11].

Gas-sensitive materials for low-temperature hydrogen sensors have been synthesized. PdO nanoparticles with added palladium allow for the detection of very high hydrogen concentrations, exceeding 1000 ppm at room temperature [12]. A nanomaterial based on WO_3, graphene, and palladium exhibited a response of several percent at a hydrogen concentration of 10000 ppm and a temperature of 50 °C [13]. The response of Ti_2CTx MXene to 1000 ppm of hydrogen was several percent [14]. A material based on TiO_2 nanospheres with palladium additives allowed for the detection of hydrogen at a concentration of 500 ppm [15]. A material based on $NiCo_2O_4$ nanoneedles with an addition of palladium allowed for the confident detection of hydrogen at a concentration of 100 ppm [16]. A nanomaterial based on WO_3 allowed for a sensor response of 12% at a hydrogen concentration of 100 ppm [17]. A nanomaterial based on mixed manganese and cobalt oxides with an addition of reduced graphene had a similar response [18]. A significantly greater response to hydrogen was observed in a tungsten (VI) oxide-based nanomaterial with a palladium additive [19]. Using TiO_2-based nanomaterials, significant responses to hydrogen were obtained at room temperature, but this required the use of UV radiation to activate the sensitive material and impedance measurements [20].

Indium (III) oxide-based nanomaterials are widely used [21, 22]. For example, silver-modified In_2O_3 nanosheets were used to detect 1-butanol under UV light [23]. Thermal spraying of thin-film indium oxide allowed for the detection of NO_2 and H_2S at room temperature [24].

An important problem in the determination of gases by low-temperature ("room temperature") semiconductor sensors is the minimization of the influence of the humidity of the environment. The presence on the surface of metal oxide semiconductors of hydroxyl groups formed during synthesis makes them vulnerable to water sorption, which leads to a significant increase in the contribution of ionic conductivity. The electroconductive response of semiconductor sensors is based on a change in the concentration of electrons and a change in their mobility, so an increase in the contribution of ionic conductivity interferes with the gas analysis. Thus, in order to obtain gas-sensitive materials for low-temperature sensors, it makes sense to choose methods in which the interaction of the reactants does not occur in an aqueous environment. Most studies on "room temperature sensors" do not use either sol-gel synthesis or hydrothermal synthesis.

In this paper, the synthesis of the In_2O_3 nanomaterial was carried out using the biotemplate method, which has been widely used in recent years for the production of biocatalysts, antibiotics, and anti-cancer drugs

[25]. Biotemplate synthesis is also used for the production of gas-sensitive materials. For example, a CeO_2-ZnO hollow-fiber-based material was successfully used to create an ethanol sensor [26]. SnO_2-based nanotubes were used to create an acetone sensor [27].

The purpose of our work was not only to study the gas sensitivity of the obtained material in relation to hydrogen, but also to study the effect of humidity on the background resistance, since this problem is not considered in publications on "room temperature sensors" for unknown reasons.

2. Experimental

2.1. Synthesis

A weighed amount of indium nitrate (CAS: 207398-97-8, InN_3O_9·nHOH, MW: 300.83 g/mol, form: powder and chunks, Sigma-Aldrich, USA), corresponding to 0.05 mol(eq)/L, was mixed with an aqueous solution of acetic acid (pH = 5). The mixture was kept at room temperature until the indium nitrate was completely dissolved.

The sheets of ash-free cellulose filters (red tape) were washed with an eluent, a mixture of butanol and acetic acid in a volume ratio of 1:4. 50 ml of the eluent was poured into a 0.5-liter glass, and the cellulose sheets were immersed in the solution to a depth of 1-2 cm. The glass was covered with glass and left for 5 hours. Next, the sheets were removed from the cup, dried, and after drying, the top 2 cm of the sheets were cut off and removed, and then heated for 3 hours in a drying oven at 105 °C.

50 ml of the solution of indium nitrate was poured into a glass, and sheets of washed and dried filter paper were immersed in it to a depth of 1 cm. The glass was closed and left to stand for 2 hours. After impregnation, the cellulose was removed from the chamber and dried at 105 °C for 3 hours. The material was then calcined at 500 °C for 6 hours to burn off the cellulose and form indium oxide:

$$4In(NO_3)_3 \rightarrow 2In_2O_3 + 12NO_2 + 3O_2 \quad (1)$$

2.2. Sensor fabrication

A gas-sensitive layer based on In_2O_3+Pd (mass fraction of palladium 3%) was created by treating the In_2O_3 material with a solution of tetraammine palladium (II) nitrate. After drying, the material was mixed with terpeniol, which was used as a binding agent, to form a paste. The resulting paste was applied to a dielectric substrate made of aluminum oxide, with platinum electrodes and a heater, and then baked at a temperature of 750 °C, resulting in the burning of the terpeniol and the formation of a semiconductor layer of brittle indium oxide gel on the substrate.

2.3. Material characterization

The structure of the In_2O_3 sample was characterized by X-ray phase analysis using a DRON-4 instrument with a cobalt anode. The resulting diffraction patterns were subsequently analyzed using the ICSD Database 2010-2).

The material was investigated using a JEOL JSM-6380LV scanning electron microscope in secondary electron mode.

2.4. Sensor measurements

Calibration gas mixtures "hydrogen in synthetic air" with concentrations of 10 ppm and 200 ppm were used. To achieve the desired hydrogen concentration, the calibration gas mixtures were diluted with synthetic air. A portion of the synthetic air flow was passed through distilled water for humidification. After mixing the three streams - dry air, humidified air, and the verification gas mixture, the humidity and temperature were measured using a Honeywell HIH-4602-A sensor. A TO-8 sensor in a metal casing was placed in a stainless-steel chamber.

Using a specially designed device, the electrical resistance of the gas-sensitive layer of the sensor was measured at a sampling rate of 40 Hz and recorded as a computer file.

The sensor response S was determined as the relative difference in electrical conductivity in the gas medium σ and in synthetic air σ_0, which is equivalent to the relative difference in electrical resistance in the gas medium R and in synthetic air R_0:

$$S = \frac{\sigma - \sigma_0}{\sigma_0} = \frac{R_0 - R}{R}. \quad (2)$$

3. Results and discussion

3.1. Morphology and structure of the material

Fig. 1 shows images of an indium oxide sample obtained using a scanning electron

microscope. The samples retain the characteristic macrostructure of the cellulose template. In terms of morphology, the studied material consists of agglomerations of fiber-like objects, each of which is curved or "twisted," sometimes multiple times, along its length, which ranges from a few micrometers to several tens of micrometers. The thickness of the relatively flat fibers can be as thin as submicrons, while the width can be several micrometers, forming extended "sheets." In some areas of fiber agglomerations, there are objects of small thickness (size) of ~ 100 nm.

As a result of the deciphering of diffraction patterns, it was found that the studied sample is almost completely (more than 95%) represented by the main modifications of crystalline indium (III) oxide, which is cubic (PG Ia). In addition, the presence of mixed indium oxide-hydroxide InO(OH) (cubic, PG P213, up to 3%) and indium hydroxide $In(OH)_3$ (cubic, PG Im, less than 1%) was detected in small amounts, which can be explained by insufficient thermal treatment in terms of time or temperature. The data on R-factors and the quality factor of the diffraction pattern are also shown in Figure 2.

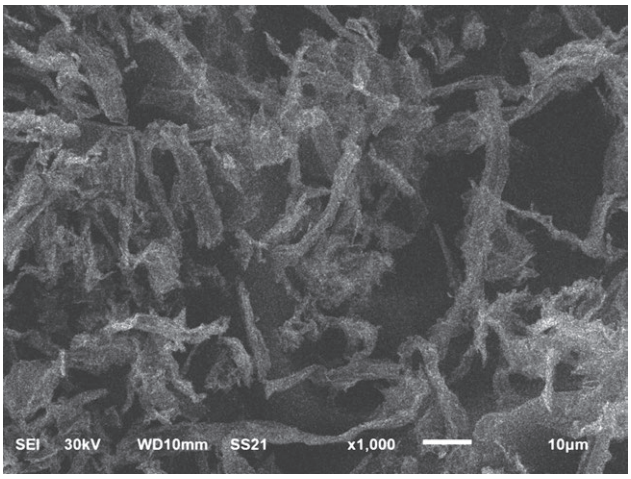

Fig. 1. SEM image of In_2O_3 material obtained by the biotemplate method

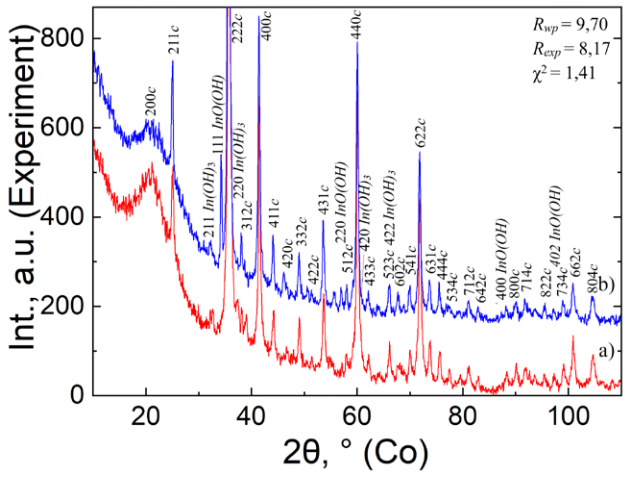

Fig. 2. X-ray diffraction pattern of indium (III) oxide nanopowder, calcinated at 500 °C. Compounds: *c* – In_2O_3 (cubic, SG *Ia*); InO(OH) (cubic, SG $P2_13$), $In(OH)_3$ (cubic, SG *Im*)

3.2. Sensor investigation

Fig. 3 shows the electrical resistance of the sensor when hydrogen is added to the air at a concentration of 10 ppm. As expected, the response is donor-type, resulting in an increase in the electrical conductivity of the sensor. This is because indium oxide is an n-type semiconductor, and hydrogen is a reducing agent. Adding hydrogen increases the concentration of electrons in the semiconductor:

$$H_2 \rightarrow 2H^+ + 2e \quad (3)$$

$$2H_2 + O_2^- \rightarrow 2H_2O + e \quad (4)$$

Fig. 4 shows the calibration dependence of the In_2O_3+Pd sensor at a temperature of 25 °C. The sensitivity of our sensor is higher than in papers [12-18].

3.3. Determining the effect of humidity

As already noted, an important problem of low-temperature sensors is to minimize

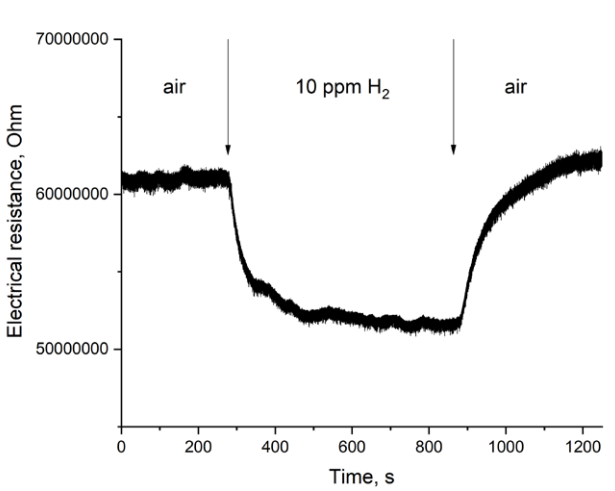

Fig. 3. Electrical resistance of the In_2O_3-Pd sensor when hydrogen is added. Sensor temperature is 25°C, the air humidity is 25 %

the influence of humidity. Fig. 5 shows the dependence of the electrical resistance of the In_2O_3-Pd sensor on humidity at a temperature of 25 °C.

As expected, an increase in humidity leads to a significant decrease in electrical resistance, which is due to the appearance of an additional (ionic) conduction mechanism caused by the sorption of water on the surface of the metal oxide semiconductor [28]. This mechanism is caused by the dissociation of water and the appearance of a significant number of hydrogen cations on the surface. Additionally, charge transfer can also be associated with the transport of hydroxide anions.

4. Conclusion

In_2O_3-Pd nanomaterial samples synthesized by the biotemporal method showed the ability to detect relatively low concentrations of hydrogen at room temperature, which suggests that they can be used to create sensors that do not require energy consumption for heating.

Despite the choice of a non-aqueous synthesis method and the minimal number of hydroxyl groups in the metal oxide semiconductor, the resistance of the resulting sensor is significantly affected by humidity, so the practical application of the corresponding gas analyzer is only possible in combination with a humidity sensor.

Contribution of the authors

The authors contributed equally to this article.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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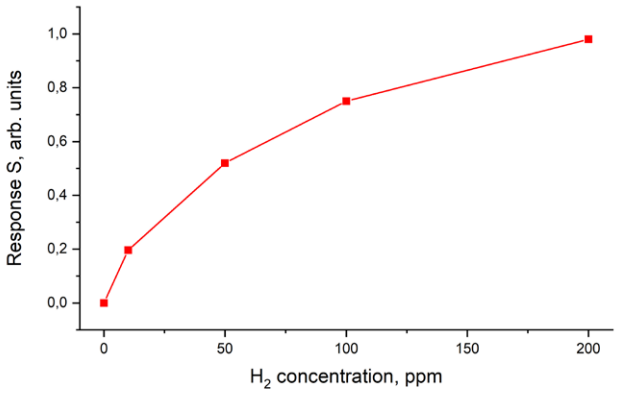

Fig. 4. Dependence of the In_2O_3-Pd sensor response on hydrogen concentration at a temperature of 25 °C

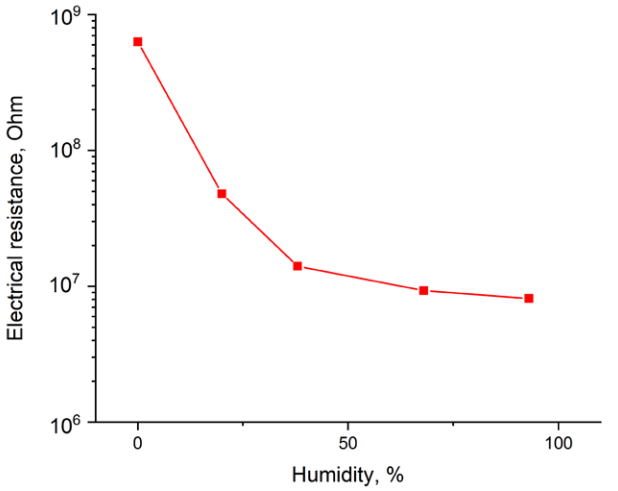

Fig. 5. Dependence of the electrical resistance of the In_2O_3-Pd sensor on air humidity at a temperature of 25 °C

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Received April 3, 2025; approved after reviewing April 21, 2025; accepted for publication May 15, 2025; published online December 25, 2025