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SYNTHESIS AND STUDY OF GAS-SENSITIVE PROPERTIES OF FILMS BASED ON TIN OXIDE

Abstract

The article looks at how to make sensitive parts for gas analyzers that work at room temperature using thin films of tin oxide (SnO_2) that are deposited on glass substrates. Three precursor systems were employed: a solution of $SnCl_4$*$5H_2O$ in ethanol, a hydrosol of tin hydroxide, and a combination of them. The films were formed by spray pyrolysis at 400°C. X-ray structural analysis and scanning electron microscopy were performed; it was found that the crystallite sizes were 6–13 nm. We studied the sensitivity of the films to water vapor. The highest sensitivity (R_0/R_{vapor} = 3.75) and response time (less than 1 second) were observed in films obtained from the sol. When adsorption on t-SnO_2 (001) and c-SnO_2 (111) surfaces were modeled, it was found that the c-SnO_2 structure is better for detecting carbon monoxide because it stays stable in high humidity. The results obtained are of interest in the development of new gas sensors

Keywords: tin oxide (SnO_2), thin films, spray pyrolysis, sensory properties, adsorption of molecules, gas analyzers.

Introduction

Tin oxide (SnO_2) thin films are among the most studied materials for gas sensing applications due to their high sensitivity, fast response, and chemical stability. As an n-type wide bandgap semiconductor, SnO_2 exhibits gas-sensitive behavior primarily through surface interactions with adsorbed species, which affect its electrical conductivity [1, 2]. These features make SnO_2 a promising candidate for environmental monitoring and industrial safety systems [3, 4].

Traditionally, SnO_2-based gas sensors operate at elevated temperatures (typically above 200 °C) to achieve sufficient sensitivity and response times. However, this imposes limitations on sensor miniaturization, energy efficiency, and long-term stability, especially in compact or wearable

devices [5]. Therefore, the development of sensitive layers that can operate effectively at room temperature remains a critical challenge in modern sensor design.

The properties of SnO_2 thin films are highly dependent on the synthesis method and precursor composition. Numerous fabrication approaches—such as magnetron sputtering, sol-gel methods, inkjet printing, and spray pyrolysis—have been explored to tailor the microstructure, porosity, and defect chemistry of the films [6, 7]. For instance, magnetron sputtering enables the formation of nanocrystalline structures with high surface area, which is favorable for gas adsorption [3]. Similarly, variations in deposition parameters have been shown to influence film thickness and, consequently, gas sensitivity [4]. The presence of oxygen vacancies and structural defects plays a key role in enhancing gas sensitivity, as they serve as active sites for molecular adsorption [1, 8].

Among the available methods, spray pyrolysis stands out for its simplicity, cost-effectiveness, and scalability, making it suitable for fabricating gas-sensitive layers on glass and flexible substrates [9]. Moreover, doping strategies and catalyst addition–such as palladium or tungsten–can further enhance sensitivity and selectivity by promoting chemical interactions with target gases [10–12]. Morever, advances in thin-film technology allow for the miniaturization and integration of these sensors into compact systems, thereby promoting wider applicability in real-world scenarios without compromising on sensitivity [13–14].

In this work, we investigate the gas-sensitive behavior of SnO_2 thin films obtained by spray pyrolysis at 400 °C using three distinct precursor systems: (1) a solution of $SnCl_4 \cdot 5H_2O$ in ethanol, (2) a hydrosol of tin hydroxide, and (3) a mixed composition of both. The structural and morphological properties of the resulting films were characterized by X-ray diffraction and scanning electron microscopy. Crystallite sizes ranged from 6 to 13 nm depending on the synthesis route.

The films were tested for their sensitivity to water vapor under ambient conditions. Among the samples, the film derived from the hydrosol exhibited the highest sensitivity (R_0/R_vapor = 3.75) and the fastest response time (less than 1 second). To complement the experimental findings, we conducted ab initio modeling of water and carbon monoxide adsorption on different SnO_2 surface terminations. The results show that the (111) surface of the cubic phase (c-SnO_2) maintains structural stability under high humidity, suggesting its potential for selective CO detection in humid environments.

Overall, the findings contribute to the design of efficient room-temperature gas sensors by demonstrating the importance of precursor chemistry and surface structure.

Materials and Methods

Tin oxide was chosen as a compound that changes resistance with changes in the composition of the environment, and a glass slide was used as a substrate.

We used three film-forming systems to create tin oxide films on a glass substrate:

1. Pentahydrate tin chloride tetrahydrate was mixed with ethanol to make a solution that had 0.11 mol/l of tin ions in it [15].

2. An aqueous solution of $SnCl_4 \cdot 5H_2O$ with 0.11 mol/l of tin ions was heated on an electric hotplate at 100˚C for 1.5 hours, or until the color changed. This was done to make hydrosol. This formed a tin hydroxide hydrosol according to the reaction:

$$SnCl_4 + 4H_2O \rightarrow Sn(OH)_4 \downarrow + 4HCl \quad (1)$$

3. A mixture of the ethanol system and tin hydroxide hydrosol in a 1:1 ratio by volume.

The film-forming systems were prepared from tin tetrachloride crystal hydrate ($SnCl_4 \cdot 5H_2O$) of the “pure” grade, rectified alcohol, and distilled water.

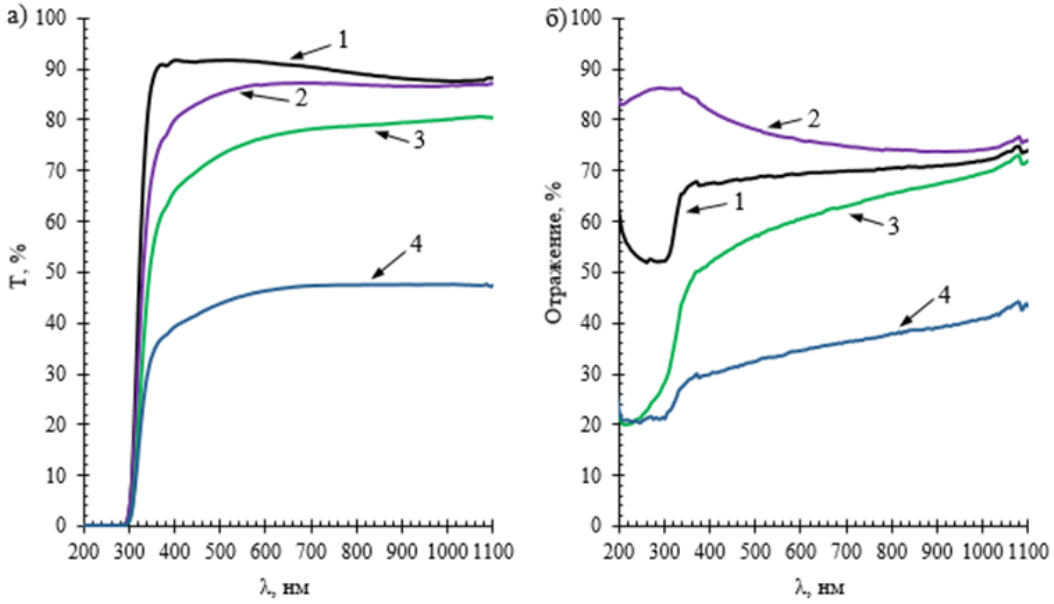
The films were applied by spray pyrolysis onto a substrate heated to 400˚C. The final reaction is the formation of tin oxide:

$$Sn(OH)_4 \; 400°C \rightarrow SnO_2 + H_2O \quad (2)$$

The structure of the films was studied using a JEOL JSM-6490LA scanning electron microscope and a DRON-6 X-ray diffractometer. The change in resistance when water vapor was introduced was measured using the four-probe method.

Results and Discussion

Figure 1 shows the transmission and reflection spectra of the samples under study. As can be seen in Figure 1(a), the transmission of the glass substrate in the visible and near-infrared regions is approximately 85–90%. A sharp decrease in the transmission of electromagnetic waves with wavelengths less than 300 nanometers corresponds to the absorption edge of glass. A sample with a film obtained from an ethanol system has a transmission of 80–85% in the same regions. This means that films produced from ethanol-based systems can be used for architectural windows and car window coatings. Samples with a film obtained from mixtures of systems and hydrosols have a visible spectrum transmission of less than 70%. According to GOST 32565-2013, paragraph 5.1.2.5 states that the light transmission of windows must be at least 70% in order to provide visibility for the driver from both the front and back. Therefore, the use of hydrosols and ethanol mixtures in the production of transparent coatings is not recommended. However, a decrease in transmission may be due to the surface structure of the film.



1 – Glass, 2 – Sample from an ethanol system, 3 – Sample from a mixture, 4 – Sample from a hydrosol
a) Transmission spectra, b) Reflection spectra

Figure 1 – Spectra of tin oxide thin films

Figure 1 (b) shows the reflection spectra of electromagnetic waves directed at a 45-degree angle to the samples normal. It can be observed that the reflection from a sample with a film produced from an ethanol-based system (Figure 1 (b), curve 2) is higher than that of a glass sample without a film (Figure 1(b), curve 1). This indicates that films produced from ethanol-based systems can be used as energy-efficient coatings, as more of the energy carried by electromagnetic waves will be reflected back into the environment.

The smallest reflection of the samples studied was observed on samples with a film obtained from a hydrosol (Fig. 5.1 (b), curve 4). This may be due to the scattering of electromagnetic radiation on the irregularities of the surface.

The thickness of the samples, determined by the change in sample mass, was 500±15 nm for all films. X-ray structural analysis was performed to determine the crystallinity of the films obtained. Table 1 presents the results of the crystallite size calculations.

Table 1 – SnO_2 crystallite sizes

Film-forming system	SnO2		
	(110)	(101)	(200)
On ethanol	13,6±0,2 nm	10,7±0,1 nm	10,5±0,3 nm
Mixture	12,4±0,1 nm	12,6±0,1 nm	10,1±0,1 nm
Sol	6,2±0,2 nm	6,3±0,2 nm	10,3±0,4 nm

Table 1 shows that all films consist of tin oxide crystallites with a size of ~6–13 nm, i.e., they are nanostructured. The literature data confirms this, stating that SnO crystals form at 400°C [16–19]. The film-forming system on ethanol directs the preferential growth of crystallites along the (110) plane [20]. Along the 101 and 200 planes, the sizes differ within the limits of measurement accuracy. The crystallites formed during the sol preparation have sizes of about 6 nm along the (110) and (101) planes. The formation of crystallites from the mixture of film-forming systems occurs in two stages. The first is the formation during the preparation of the hydrosol. Next is the growth of the crystallites that were made in the first step, which is caused by ions from the system that form the film on ethanol. As as it can be seen in Table 1, the crystallites that form in the film when different film-forming systems are mixed grow along the planes (110) and (101). The sizes of crystallites are associated with sensitivity to gases in the environment [21–24].

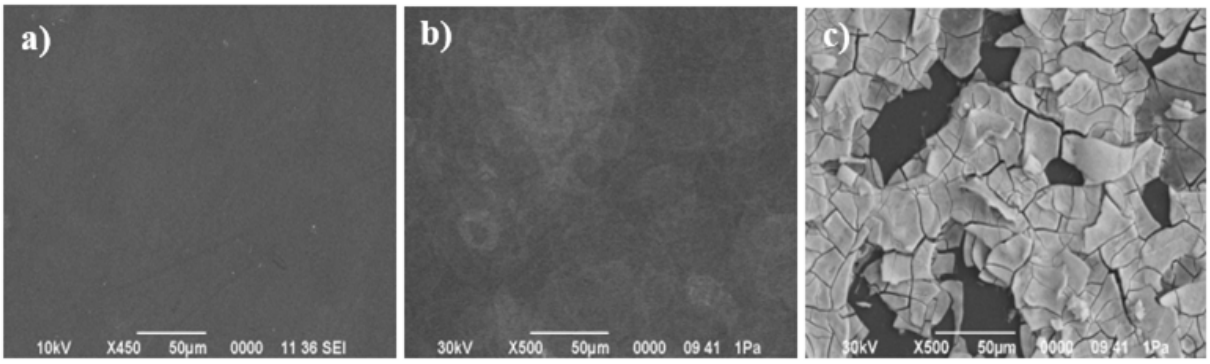
Table 2 presents the study's results on samples' sensitivity to water vapor at room temperature (22 °C).

Table 2 – Parameters of the sensitive layer of samples

Parameters	Sample		
	On ethanol	Mixture	Sol
Initial resistance value R0	9,7±0,4 kOhm/sq.	140±5 MOhm/sq.	150±5 MOhm/sq.
The lowest value of resistance when water vapor is released Rvapor	9,7±0,4 kOhm/sq.	52±1,5 MOhm/sq.	40±1,5 MOhm/sq.
Sensitivity R0/Rvapor	-	2,69	3,75
Response time (speed) tresponse	-	4-5 sec.	Less than 1 sec
The time to restore the initial resistance value trestore	-	10-12 sec.	≈ 3 sec.

Table 2 shows that the film from the ethanol system is not sensitive to water vapor. The film from the sol exhibits sensitivity to ethanol vapor. When water vapor is introduced, it changes its resistance by 3,75 times in less than 1 second. It restores its initial resistance value in about 3 seconds. Films obtained from a mixture of film-forming systems are also sensitive to water vapor.

Based on the SEM images of the film surface shown in Figure 2, we can estimate the effect of the composition of the film-forming system on the morphology of the surface of films formed on a glass substrate.



a) synthesized from an ethanol system; b) synthesized from a mixture of systems; c) synthesized from sol.

Figure 2 – SEM images of the film surface

As can be seen in Figure 2(a), the film obtained from the ethanol system has a continuous and homogeneous structure. Similarly, the film produced from the mixture in Figure 2(b) also has a consistent structure. However, there are individual teardrop-shaped spots caused by the application process. In contrast, the film made from sol in Figure 2c is cracked and has weak adhesion to the substrate, flaking off at room temperature. Despite its high-water vapor sensitivity of 3.75 arb. units (Table 2), it is also prone to partial crumbling when in contact with foreign objects.

However, the response time and the time of restoration of the initial resistance value are longer than those of films obtained from the sol. Thus, obtained tin oxide films on a glass substrate that are sensitive to water vapor at room temperature. Supplying water vapor causes a 3.75-fold change in the resistance of the films. The response time is less than 1 second; the restoration time is about 3 seconds. At room temperature, films made from a 1:1 mix of hydrosol and ethyl solution of tin tetrachloride are also sensitive to water vapor. At the same time, they are more resistant to abrasion

Adsorption modeling

Two model systems were used to model thin tin films. The first is a 1.5 nm thick layer of tetragonal tin dioxide with a (001) surface. The second structure corresponds to cubic tin dioxide's (111) surface (hereinafter t-SnO_2 and c-SnO_2). The first structure corresponds to films grown on substrates with cubic symmetry in the surface layer, and the second on substrates with hexagonal symmetry. This type of surface is the main one for many metals with fcc lattices, such as copper. Firstly, the energies of vacancy formation and added an additional oxygen atom were calculated. The calculation results show fairly high energies of vacancy formation (see Table 3), corresponding to temperatures above 200 °C. At the same time, the energies required for oxygen addition are very low. Because all vacancies in the surface layer will oxidize quickly, we excluded structures with vacancies from consideration.

Next, the adsorption of various molecules on these surfaces were simulated. The calculation results presented in Table 3 show that oxygen, carbon monoxide, and water will stably deposit on the (001) surface of t-SnO_2 at room temperature, while only carbon monoxide and water will deposit on the (111) surface of c-SnO_2. It should be noted that carbon monoxide and water occupy the same positions on the (001) surface of t-SnO_2 and different positions on the (111) surface of c. Thus, all available positions on the t-SnO_2 surface will be occupied by oxygen, and, under high humidity conditions, by water, as evidenced by the Langmuir isotherms shown in figure 1. While on the c-SnO_2 surface, water and carbon monoxide will occupy different active positions, even at minimal pressure (see Figure 1). Based on these results, it can be concluded that the (111) surface is suitable for use in carbon monoxide sensors.

Table 3 – Calculated values of the energy of defect formation and adsorption of molecules on the discussed tin dioxide surfaces

Defect	Energy of formation (kJ/defect)			
	Tetragonal SnO_2 (001)		Cubic SnO_2 (111)	
vO +O	+181.9 +89.3		+204.5 +14.6	
Molecules	Enthalpy / Free energy of adsorption at room temperature (kJ/mol)			
NO_2 CO_2 O_2 H_2O $4H_2O$ $8H_2O$ CO $+4H_2O$ $+8H_2O$	-37.9 +2.9 -44.0 -82.1 -27.9	-4.8 +19.0 -32.5 -50.8 -8.5	+371.8 +17.2 -1.3 -109.4 -119.3 -122.3 -66.5 -65.7 -74.0	+404.9 +33.3 +10.2 -78.1 -88.0 -91.0 -47.2 -46.3 -54.7

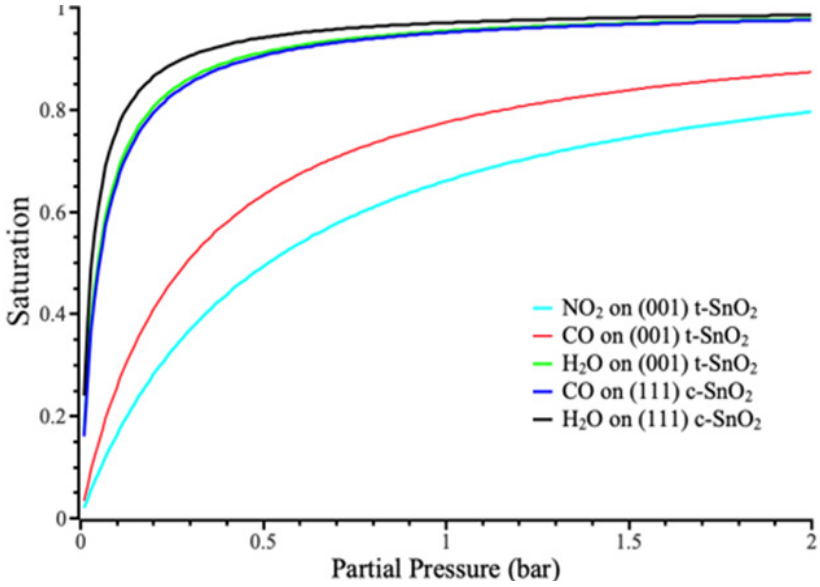


Figure 3 – Langmuir isotherms for gas adsorption on the studied surfaces

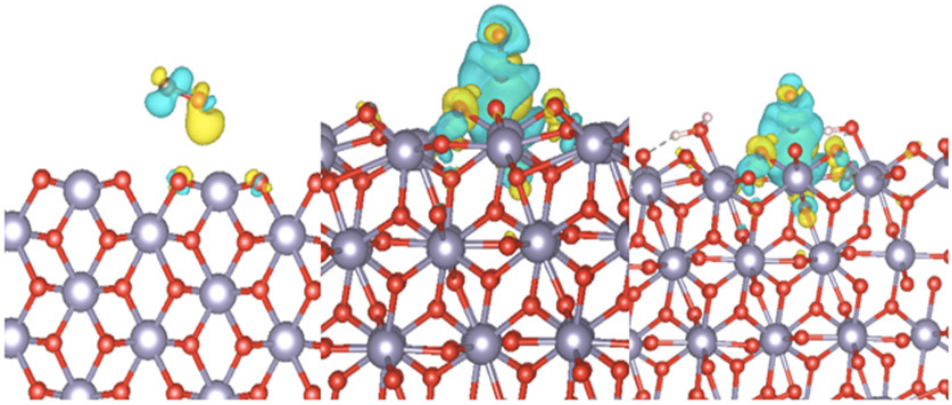


Figure 4 – Changes in charge density for the cases of carbon monoxide molecule adsorption on the (001) surface of t-SnO_2 (left panel) and on the (111) surface of c-SnO_2 for the cases of low (center panel) and high (right panel) humidity. Blue and yellow "clouds" correspond to a decrease and increase in charge density

The final stage of this work was to evaluate the effect of carbon monoxide adsorption on the substrate. This step allows to evaluate the effect of charge transfer from the molecule to the substrate, which is essential for assessing the suitability of the material as a sensor. As can be seen from figure 2, carbon monoxide adsorption on the (001) surface of t-SnO_2 leads to a visible charge redistribution in the molecule itself and insignificant doping of SnO_2, small yellow "clouds". In contrast, carbon monoxide molecule adsorption on the (111) surface of c-SnO_2 leads to a serious redistribution of the charge density in the surface layers and a visible change in the subsurface layers. Considering that the surface (111) is likely to be saturated with water molecules, the change in charge density was also calculated for the case of high humidity. Figure 2 shows that the pattern of charge density redistribution remains unchanged, even at an H_2O:CO ratio of 8:1. Thus, the sensor properties of the (111) surface of c-SnO_2 will be unchanged in dry and humid atmospheres.

Conclusion

In this work, a comprehensive experimental and theoretical investigation of the sensor properties of tin oxide (SnO_2) films was conducted to evaluate their potential for use in gas analyzer systems. SnO_2 films synthesized from hydrosol precursors demonstrated high sensitivity to water vapor at room temperature, with notably fast response (less than 1 second) and recovery times (approximately 3 seconds). These characteristics are particularly valuable for real-time environmental monitoring and safety systems that require immediate detection of hazardous or critical gas concentrations. Furthermore, the use of mixed hydrosol/$SnCl_4 \cdot 5H_2O$ systems resulted in improved mechanical robustness and resistance to surface abrasion, which is a crucial factor in ensuring sensor longevity under industrial operating conditions.

The modeling component of the study provided key insights into the molecular-level interactions at the surface of different SnO_2 polymorphs. It was found that the (111) surface of cubic SnO_2 (c-SnO_2) retains its ability to detect carbon monoxide even under high humidity conditions, due to favorable redistribution of charge density across surface and subsurface atomic layers. This finding is particularly significant, as it addresses a common challenge in gas sensing applications – signal degradation due to water vapor interference.

The results of this study contribute directly to the development of next-generation gas sensors designed for low-temperature, moisture-rich environments, such as underground mining operations, chemical processing facilities, and smart building air quality monitoring systems. The demonstrated combination of high sensitivity, rapid response, structural durability, and moisture-tolerant selectivity makes these materials promising candidates for explosion-proof sensor platforms, where rapid detection and operational reliability are critical.

Future work will focus on integrating the synthesized films into miniaturized sensor devices, validating their performance in real-world environments, and optimizing device architecture based on the surface interactions revealed through modeling. Overall, the synergy between synthesis, experimental characterization, and atomistic modeling in this study provides a solid foundation for the rational design of SnO_2-based sensor materials tailored to specific industrial and safety applications.

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ҚАЛАЙЫ ОКСИДІ НЕГІЗІНДЕГІ ЖАБЫНДАРДЫҢ ГАЗҒА СЕЗІМТАЛ ҚАСИЕТТЕРІН ЗЕРТТЕУ ЖӘНЕ СИНТЕЗДЕУ

Андатпа

Мақалада бөлме температурасында жұмыс істейтін газ анализаторлары үшін сезімтал элементтер жасау мақсатында шыны төсеніш негізінде жасалатын қалайы оксидінің (SnO_2) жұқа қабықшалары зерттелген. Ол үшін үш жабын түзетін жүйе қолданылды: этанолдағы $SnCl_4 \cdot 5H_2O$ ерітіндісі, қалайы гидроксиді гидрозолі және олардың қоспасы. Жабындар 400 °C температурада спрей-пиролиз әдісімен қалыптастырылды. Рентгендік құрылымдық талдау және сканерлеуші электрондық микроскопия жүргізілді, кристаллиттердің өлшемдері 6–13 нм екендігі анықталды. Жабындардың су буына сезімталдығы зерттелді. Ең жоғары сезімталдық ($R_0/Rбу = 3,75$) және өнімділік (1 секундтан аз) золадан алынған жабындарда байқалды. t-SnO_2 (001) және c-SnO_2 (111) беттеріндегі адсорбцияны модельдеу жоғары ылғалдылық жағдайында тұрақты сенсорлық қасиеттеріне байланысты c-SnO_2 құрылымы көміртегі тотығын анықтауға қолайлы екенін көрсетті. Алынған нәтижелер жаңа газ сенсорларын жасауға қызығушылық тудырады.

Тірек сөздер: қалайы оксиді (SnO_2), жұқа қабықшалар, бүріккіш пиролиз, сенсорлық қасиеттер, молекулалық адсорбция, газ анализаторлары.

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СИНТЕЗ И ИССЛЕДОВАНИЕ ГАЗОЧУВСТВИТЕЛЬНЫХ СВОЙСТВ ПЛЕНОК НА ОСНОВЕ ОКСИДА ОЛОВА

Аннотация

В статье исследованы тонкие пленки оксида олова (SnO_2), полученные на стеклянных подложках, с целью создания чувствительных элементов для газоанализаторов, работающих при комнатной температуре. Были использованы три пленкообразующие системы: раствор $SnCl_4$*$5H_2O$ в этаноле, гидрозоль гидроксида олова и их смесь. Пленки формировались методом спрей-пиролиза при 400 °C. Проведены рентгеноструктурный анализ и сканирующая электронная микроскопия, установлено, что размеры кристаллитов составляют 6–13 нм. Исследована чувствительность пленок к парам воды. Наиболее высокая чувствительность ($R_0/R_{пар}$ = 3,75) и быстродействие (менее 1 секунды) наблюдаются у пленок, полученных из золя. Моделирование адсорбции на поверхностях t-SnO_2 (001) и c-SnO_2 (111) показало, что структура c-SnO_2 перспективна для детектирования угарного газа благодаря стабильным сенсорным свойствам в условиях высокой влажности. Полученные результаты представляют интерес для разработки новых сенсоров газов.

Ключевые слова: оксид олова (SnO_2), тонкие пленки, спрей-пиролиз, сенсорные свойства, адсорбция молекул, газоанализаторы.

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