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GAS SENSITIVE ELEMENTS BASED ON ORGANIC-INORGANIC NANOCOMPOSITES

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Background. For today there is high need of cheap portable gas sensors for operational monitoring of the environment and the atmosphere in different areas of life and industry. Recently, hybrid nanosystems based on conductive polymers reinforced with semiconductor nanoparticles of different nature are in the focus of increased attention as materials for sensor elements.

Objective. Creating sensitive elements based on composite films of poly-3,4-ethylenedioxythiophene combined with nanocrystals of porous silicon and zinc oxide and studying the electrical response to the absorption of gas molecules.

Methods. The structure of ZnO nanoparticles and porous silicon powder was examined with X-ray diffraction. Organic-inorganic hybrid films were characterized by scanning electron microscopy and Fourier transform infrared spectroscopy. To evaluate the sensor properties, electrical response of obtained composite films due to adsorption of ammonia and ethanol molecules were studied.

Results. Our studies suggest some interaction between organic and inorganic components in the formed hybrid monolithic film. Increasing of nanocomposite electrical resistance due to adsorption of ammonia and ethanol molecules was registered. It was established that the maximum sensitivity of the hybrid films is observed at low concentration ranges. The kinetics of the response of the hybrid composites to the changing concentration of gas molecules is fast enough to be employed in various microelectronic chemical sensors.

Conclusions. The combination of the porous silicon and zinc oxide nanoparticles provides an increasing of surface area of the sensors based on organic-inorganic composites and their high sensitivity and selectivity to ethanol and ammonia molecules. **Keywords:** sensor; nanocomposite; conducting polymer; porous silicon; zinc oxide.

Introduction

For today there is high need of cheap portable sensors for operational observation of environmental and industrial processes, for monitoring of gaseous media in food industry, especially, control of food freshness in the process of the storage and other purposes. Creation of chemical sensors is an important to monitor the environment, the atmosphere of residential and office spaces, quality of drinks and medicines in the different areas of life and industry [1,2]. It is necessary to note the prospects of using thinfilm sensors based on organic compounds among all the existing sensor materials and devices. Novel organic materials for gas sensing devices and molecular electronics have been developed on the basis of conducting polymers [3-5]. Recently, hybrid nanosystems on the base of conductive polymers reinforced with inorganic nanoparticles of different nature are in the focus of increased attention as materials for sensor elements [6-8]. Film sensors based on the organicinorganic nanocomposites do not require high operating temperatures, are simple in design and can be manufactured by low-cost methods.

Particularly interesting example of a conducting polymer with conjugated backbone and controlled electron characteristics is poly(3,4-ethylene-

dioxythiophene) abbreviated hereafter as PEDOT owing to its remarkable optical and electrical properties. Due to doping-dedoping processes, the electronic properties (e.g., the bandgap) of the conjugated polymers can be varied substantially [9–11]. In addition, it has been shown that adsorption of gas molecules such as CO [12] and NH₃ [8], as well as vapors of organic solvents [13] or water molecules [7], can strongly affect physical and chemical characteristics of PEDOT and the relevant composites.

Incorporation of low-dimensional components such as carbon, silica and ZnO nanoparticles gives a possibility to use redox-activity of conjugated polymers, size effects and large area surface of nanostructures.

A special place among the nanosized semiconductors belong to zinc oxide due to several favorable properties, including good transparency, high electron mobility, and strong room-temperature luminescence [14,15]. At the same time, one of the most important and useful application of ZnO and its nanocomposites is the manufacturing of the semiconductor sensor elements sensitive to gases both organic and inorganic nature including ethanol [16], ammonia [17] and hydrogen [18].

Also one can use silicon nanoparticles, which have high abilities to adsorption of gas molecules, for enhancing the performance of sensors [6]. Silicon nanoclusters can be prepared using a straightforward procedure of electrochemical etching of single-crystalline silicon, with further formation of a layer of "porous silicon" (PS) [19-21]. The developed surface and high surface sensitivity of PS make this material extremely perspective exactly in the field of sensor electronics [22,23].

As follows, nanostructures of PS and ZnO exhibit a significant dependence of the electrical and luminescent properties on the adsorption of molecules of different gases or vapors of organic substances and cause considerable interest as sensitive elements of chemical and biological sensor devices. Therefore, present work focuses on the creating of sensor element based on the PEDOT film doped by semiconductor nanoparticles and studying the electrical response to the absorption of ammonia and ethanol molecules.

Experiment

To obtain hybrid films was used 1.3% aqueous dispersion of polymeric complex of PEDOT doped by poly(styrenesulfonate) (PSS). A polymeric anion PSS acts simultaneously as an acid dopant and an anionic surfactant which stabilizes dispersion of the polymer [11,13]. The chemical formula of PEDOT:PSS is shown in Fig. 1. The other components of hybrid composite were ZnO and PS nanoparticles. The aqueous polymer suspension PEDOT:PSS and ZnO nanocrystals were purchased from Sigma-Aldrich Co, USA.

Fig. 1. The chemical formula of PEDOT:PSS

The PS layers were prepared by means of photoelectrochemical etching performed in galvanostatic mode on single-crystalline silicon substrates n-type conductivity with the specific resistance of 4.5 Ohm·cm. Ethanol solution of hydrofluoric acid (the volume ratio of the components HF:C₂H₅OH = 1:1) was used as an electrolyte.

The anodic current density was equal to 30 mA/cm² and the etching time was 20 min. To ensure availability

of holes in the surface layer of *n*-Si, which were necessary for formation of the PS layer, the working surface of a silicon plate was irradiated with white light during the whole process of electrochemical etching [19]. After cleaning of samples with distilled water, a resulting porous layer had been taken off from the surface of the plate. It had the shape of a finely-dispersed powder. The silicon particle sizes were ranged from a few tens of nanometers to several microns.

The structure of ZnO nanoparticles and PS powder was examined with X-ray diffraction (XRD). Data were collected on automatic diffractometer STOE STADI P (transmission mode, $2\theta/\omega$ -scan, Cu $K\alpha_1$ radiation). A preliminary data processing, X-ray profile and phase analyses were performed using the STOE WinXPOW (version 2.21) program package and by the method described in [24]. Experimentally obtained powder diffraction pattern is presented in Fig. 2.

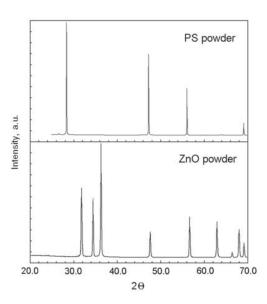


Fig. 2. X-ray diffractogram of ZnO nanocrystals and PS powder

The XRD pattern of ZnO shows a hexagonal wurtzite structure. Parameters of ZnO elementary cell are a = b = 3.2471 Å, c = 5.2019 Å, which is in a good agreement with literature [14]. An average apparent size of crystallites (size of nanoparticles) D = 52.7 nm. PS powder characterized space group Fd3m. The lattice constant and average size of coherent scattering domains are a = 5.4308 Å and D = 183.4 nm, respectively.

The obtained PS powder was mixed with nanoparticles of ZnO and the PEDOT:PSS solution and subjected to ultrasonic processing for 8 hours. After this, suspension was applied to a glass substrate and

dried at room temperature for 72 hours. Eventually, the monolithic film of PEDOT:PSS-PS-ZnO hybrid composite with PS and ZnO nanoparticles at volume ratio of 1:1 was obtained. The thickness of the film was near 20 µm.

PEDOT:PSS-PS-ZnO hybrid films were characterized by scanning electron microscopy (SEM) REMMA-102-02 ("Selmi", Ukraine) and Fourier spectroscopy. infrared (FTIR) transmittance spectra were measured with an "Avatar" spectrometer in the wave number region of 400-4000 cm⁻¹. To obtain the IR spectra of the hybrid films, the composite PEDOT:PSS-PS-ZnO was deposited on a silicon wafer with the thickness of 400 µm, using the method described above. The absorption bands of the silicon substrate were easily identified (see [25]).

In order to study sensor properties of the PEDOT:PSS-PS-ZnO hybrid composite, silver contacts were thermally deposited onto the films surface. The thickness of contacts was about 0.5 μ m. The distance between contacts was about 4 mm. The scheme of experimental samples is presented in Fig. 3.

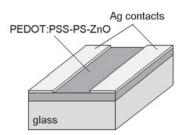


Fig. 3. The scheme of experimental sensor element

The adsorption processes in sensor element based on the PEDOT:PSS-PS-ZnO hybrid films were studied in an airtight chamber, where gas medium can be changed. The electrical resistance of our composite films was measured in the DC regime.

Results and Discussion

Analysis of the hybrid film surface was carried out using SEM methods in modes of secondary electrons and energy-dispersive X-ray spectroscopy (EDS). As one can see from Fig. 4, the PEDOT:PSS-PS-ZnO composite formed a monolithic polymer film. Study of the surfaces exhibit a considerable variation in dispersity of PS powder, mixed with nanoparticles of ZnO. A mixture of semiconductor nanoparticles was integrated into the polymer films. Developed surface of the PEDOT-PS-ZnO films extends the prospect of application of the organic-inorganic composite for gas sensing. Increasing of the working surface of the touch element enhances its sensitivity.

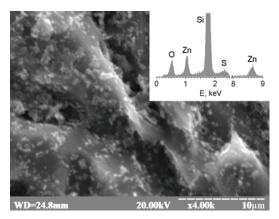


Fig. 4. SEM images and EDS of the surfaces of PEDOT:PSS-PS-ZnO film

Determination of the chemical composition of hybrid films was done based on the interpretation of energy spectra in X-ray microanalysis mode. Along with the intensive maximum at 1.74 keV, which is characteristic for silicon, peaks at 1.05 and 8.65 keV corresponding to zinc atoms were observed (see Fig. 4). The X-ray microanalysis of the hybrid film structures found the traces of carbon, oxygen and sulfur, which are components of the PEDOT:PSS polymer.

To identify the components of the PEDOT:PSS-PS-ZnO hybrid films, FTIR spectra have been measured (Fig. 5). Most intense are IR bands in the 620–660 cm⁻¹ range corresponding to bending Si-H₂ mode and the absorption band located at 1100 cm⁻¹ that can be related to valence Si-O-Si vibrations of the silicon substrate and PS nanoparticles [25,26]. The band at 460 cm⁻¹ is usually ascribed to deformation vibrations of Si-O group [25]. In general, the bands are observed due to oxidation of the silicon surface and adsorption of the water molecules from the atmosphere.

The absorption bands observed in the FTIR spectra of the PEDOT:PSS-PS-ZnO composite, which are located in the regions 1080–1310 and 1500–1550 cm⁻¹, are characteristic for C-O, Si-O-C complexes and C=C, C-C vibrations of thiophene rings [11, 27]. The absorption band at 700 cm⁻¹ can be ascribed to valence vibrations C-S [7].

Besides, the IR spectra of the hybrid composite include the absorption band with the maximum located at 860 cm^{-1} and the peaks in the regions of 950– 1000 cm^{-1} (see Fig. 5). These bands are characteristic for hydrogen containing molecular complexes: $O-SiH_x$ (x=1,2) and C-H, respectively [7,11]. The band at around 2300 cm⁻¹ usually corresponds to stretching complexes which include hydrogen, particularly, O_3-Si-H . In investigated spectral range, we did not observe absorption bands associated with molecular complexes which include zinc.

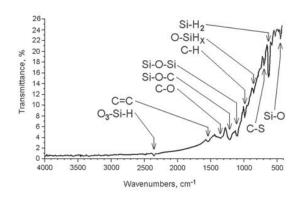


Fig. 5. FTIR spectra of hybrid PEDOT:PSS-PS-ZnO film on the silicon substrate

In the range 3400–4000 cm⁻¹, we have observed the low intensity absorption bands induced by the hydroxyl groups from Si–OH and by the adsorbed water molecules. It is worthwhile that the hydrophilic properties of the hybrid composites PEDOT:PSS–PS–ZnO expand their prospects as working elements for the sensors of humidity and other chemical compounds which contain hydrogen.

We have found experimentally that the electrical characteristics of the PEDOT:PSS-PS-ZnO films are strongly dependent on the surrounding atmosphere. In particular, increasing concentration of ammonia or ethanol molecules results in significant increase of the electrical resistance of our sensor elements (Fig. 6). The hybrid film had almost linear dependence of electrical resistance on the concentration of NH₃ molecules in the range of 1-10%, which is a significant advantage in developing of gas sensors. In the case of adsorption of C₂H₅OH molecules sublinear dependence of PEDOT:PSS-PS-ZnO film resistance the on concentration was observed.

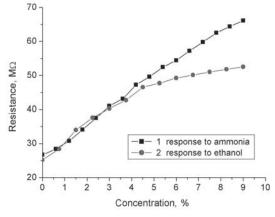


Fig. 6. Resistance of PEDOT:PSS-PS-ZnO film as functions of the ammonia (1) and ethanol (2) concentration

The character of obtained dependences caused by the interaction between polar molecules of analyzed gases and the surface of the PEDOT:PSS-PS-ZnO composite. In the case of the composite the both adsorption-induced changes in semiconductor nanoparticles and polymer should be considered. Redox processes in conjugated polymers which are caused by adsorbed molecules act as an additional doping of PEDOT: PSS.

Beside this, due to adsorption-electrical effects the electronic parameters of semiconductor nanocrystals are changed [23]. The changes occur in the polymer as well as in PS and ZnO nanoparticles contributing simultaneously to the total electrical response of hybrid PEDOT:PSS-PS-ZnO film.

An important factor of studying the mechanisms of variation of the physical parameters of sensor materials under adsorption—desorption interactions with gaseous media is determination of the sensing ability of the material. To estimate the sensing (i.e., gas-sensitive) properties of the hybrid composite PEDOT:PSS—PS—ZnO films, we have calculated their 'sensing ability' using the known relation [28]

$$\gamma = \frac{1}{R} \frac{\Delta R}{\Delta c},\tag{1}$$

where $\Delta R/R$ denotes the relative change in the electrical resistance of the hybrid film and Δc is the change in the concentration of gas molecules. The calculated dependences of sensitivity for the film sensor element on the ammonia and ethanol molecules are shown in Fig. 7.

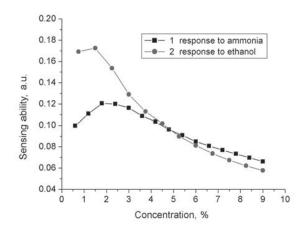


Fig. 7. Sensing abilities of resistive sensor based on the PEDOT:PSS-PS-ZnO film as functions of the ammonia (1) and ethanol (2) concentration

Based on the calculated concentration dependence of sensitivity there was found that the film sensor has a maximum adsorption sensitivity in the range of gas concentration 0.5–2.5%. In the case of the small concentrations of analyzed gases the sensitivity of the sensor is higher to molecules of ethanol and at higher concentrations – to ammonia molecules.

Considering the high sensitivity of PS to ammonia [22] and ZnO nanostructures to ethanol [16], the use of composite materials with semiconductor nanoparticles provides not only an increase in surface area of sensor elements, but also high sensitivity and selectivity to the analyzed gases. To manage functional parameters of the film sensors, the ratio between the content of ZnO and PS nanocrystals in the hybrid composite can be changed.

The dynamic dependences of response for the sensor elements based on the PEDOT:PSS-PS-ZnO film are shown in Fig. 8. The interaction with the gas molecules has a character of physical adsorption. It represents an inverse process with low activation threshold. This hypothesis is substantiated by the fact that the initial conductivity of the hybrid films is restored after scavenging and pumping off the analyzed gases from the experimental chamber.

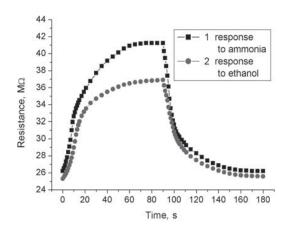


Fig. 8. Electrical resistance response of the sensor based on PEDOT:PSS-PS-ZnO film to the pulse of ammonia (1) and ethanol (2) concentration

The time of response of the sensory elements to changing gas concentration is about 60 s. Compared to the moisture-sensitive structures PS-silicon substrate [29], the response of our hybrid film sensors is much more (2–3 times) faster.

Conclusions

By very inexpensive and convenient technology we have created flexible sensitive elements based on the PEDOT:PSS-PS-ZnO composite films. Based on the analysis of SEM images of the surface of the hybrid composite was revealed a variation in dispersity of PS and ZnO powder, integrated into the polymer film. We

assume some interaction between the conjugated polymer and semiconductor nanoparticles based on the characterization of PEDOT: PSS-PS-ZnO hybrid films by SEM and FTIR spectroscopy.

The combination of the porous silicon and zinc oxide nanoparticles provides an increasing of surface area of the sensors and their high sensitivity and selectivity to ethanol and ammonia molecules. It was shown that the adsorption of analyzed gases increases the electrical resistance of PEDOT:PSS-PS-ZnO films. Based on experimental data a dependence of adsorption sensitivity of the sensor elements on the concentration of ammonia and ethanol was calculated. It was established that the maximum sensitivity of the hybrid films is observed at low concentration ranges.

The kinetics of the response of the hybrid composites PEDOT:PSS-PS-ZnO to the changing gas concentration is fast enough to be employed in various microelectronic chemical sensors. Finally, the results obtained in the present investigation would allow creation of effective gas analyzers.

Acknowledgments

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Газочутливі елементи на основі органо-неорганічних нанокомпозитів

Проблематика. На сьогоднішній день існує висока потреба в дешевих портативних газових сенсорах для оперативного моніторингу навколишнього середовища та атмосфери житлових приміщень, а також, у різних галузях промисловості. Останнім часом гібридні наносистеми на основі провідних полімерів, легованих напівпровідниковими наночастинками різної природи знаходяться в центрі підвищеної уваги як матеріали для сенсорних елементів.

Мета досліджень. Створення сенсорних елементів на основі композитних плівок полі-3,4-етилендіоксітіофену в поєднанні з нанокристалами поруватого кремнію та оксиду цинку і вивчення відгуку їх електричного опору на адсорбцію молекул газів різної природи.

Методика реалізації. Структура наночастинок ZnO та порошку поруватого кремнію була досліджена методом рентгенівської дифракції. Характеристики органо-неорганічних композитних плівок були визначені за допомогою скануючої електронної мікроскопії та IЧ Фур'є-спектроскопії. Для оцінки сенсорних властивостей був досліджений електричний відгук отриманих композиційних плівок на адсорбцію молекул аміаку та етанолу.

Результати досліджень. Експериментальні дослідження виявили взаємодію між органічними і неорганічними компонентами нанокомпозиту і утворення монолітної гібридної плівки. Зареєстровано збільшення електричного опору сенсорних елементів внаслідок адсорбції молекул аміаку і етанолу. Встановлено, що максимальна чутливість гібридних плівок знаходиться при низьких концентраціях досліджуваних речовин. Кінетика відклику резистивних сенсорних елементів на основі гібридних композитів на зміну концентрації газів є достатньо швидкою для мікроелектронних хімічних сенсорів.

Висновки. Поєднання наночастинок поруватого кремнію та оксиду цинку забезпечує збільшення площі робочої поверхні сенсорів на основі плівок органо-неорганічних композитів, їх високу чутливість і селективність до молекул аміаку та етанолу.

Ключові слова: сенсор; нанокомпозит; провідний полімер; поруватий кремній; оксид цинку.

Оленич И.Б., Циж Б.Р., Аксиментьева Е.И., Горбенко Ю.Ю.

Газочувствительные элементы на основе органо-неорганических нанокомпозитов

Проблематика. На сегодняшний день существует высокая потребность в дешевых портативных газовых сенсорах для оперативного мониторинга окружающей среды и атмосферы жилых помещений, а также в различных отраслях промышленности. В последнее время гибридные наносистемы на основе проводящих полимеров, легированных полупроводниковыми наночастицами различной природы находятся в центре повышенного внимания как материалы для сенсорных элементов.

Цель исследований. Создание сенсорных элементов на основе композитных пленок поли-3,4-этилендиокситиофена в сочетании с нанокристаллами пористого кремния и оксида цинка и изучение отклика их сопротивления на адсорбцию молекул газов разной природы.

Методика реализации. Структура наночастиц ZnO и порошка пористого кремния исследована методом рентгеновской дифракции. Характеристики гибридных органо-неорганических пленок определены при помощи сканирующей электронной микроскопии и ИК Фурье-спектроскопии. Для оценки сенсорных свойств исследован электрический отклик полученных композиционных пленок на адсорбцию молекул аммиака и этанола.

Результаты исследований. Экспериментальные исследования обнаружили взаимодействие между органическими и неорганическими компонентами нанокомпозита и образование монолитной гибридной пленки. Зарегистрировано увеличение сопротивления сенсорных элементов в результате адсорбции молекул аммиака и этанола. Установлено, что максимальная чувствительность гибридных пленок наблюдается при низких концентрациях исследуемых молекул. Кинетика отзыва резистивных сенсорных элементов на основе гибридных композитов на изменение концентрации молекул газов является достаточно быстрой для микроэлектронных химических сенсоров.

Выводы. Сочетание наночастиц пористого кремния и оксида цинка обеспечивает увеличение площади рабочей поверхности сенсоров на основе пленок органо-неорганических композитов, их высокую чувствительность и селективность к молекулам аммиака и этанола.

Ключевые слова: сенсор; нанокомпозит; проводящий полимер; пористый кремний; оксид цинка.