THE EFFECT OF UV LIGHT IRRADIATION ON THE GAS-SENSING PROPERTIES OF THE QUARTZ CRYSTAL MICROBALANCE SENSOR COMBINED WITH ZnO FILM

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Zinc oxide thin films were deposited on the both electrodes of the commercial quartz resonator HC49U using the standard radio-frequency magnetron sputtering method in order to provide NH_3, H_2O_2 and C_2H_5OH vapor sensors application at room temperature. For the first time, the impact of the ultraviolet light-emitting diode illumination (λ_{max} = 395 nm, light irradiance of about 2 mW/cm^2) on the main characteristics of the ZnO coated quartz crystal microbalance sensor, such as response/recovery time and sensitivity, was studied. The obtained experimental data and calculated characteristics prove that ultraviolet illumination of the quartz crystal microbalance sensor covered with a ZnO film leads to a significant reduction in the recovery time at detection of ethanol, ammonia or hydrogen peroxide vapors. Under the influence of ultraviolet irradiation, the fabricated quartz crystal microbalance sensor coated with ZnO film showed promising potential in detecting explosive components.

Key words: UV LED irradiation, zinc oxide, gas sensor, thin film, quartz crystal microbalance.

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I. INTRODUCTION

In recent years, there is a big boom in the sensors market. The calculated worldwide value of the sensor market is expected to reach $1 trillion by 2025 [1]. Zinc oxide is one of the first metal oxides used as a thin film material for the gas sensors due to a significant change in its conductivity in the medium of hydrogen and oxygen-containing gases. For the first time, the change in the conductivity of ZnO in the gas environment was observed in 1959, but this fact did not get enough attention [2].Thus, the first gas sensors based on the oxide materials appeared only in 1961–1962 [3]. The gas sensors based on ZnO attract considerable attention due to their high sensitivity, chemical resistance, non-toxicity and low cost [4]. About 10 % of all gas-oxide semiconductor sensors world-wide are the zinc oxide-based devices [5]. Numerous gas-sensing techniques, such as resistive/chemiresistive, electrochemical, optical, acoustic, capacitive, and quartz crystal microbalance (QCM) techniques, have been reported [1, 5–7]. Compared with other sensors, the advantages of QCM gas sensors are the high sensitivity, capability of operating at room temperature, simple technological implementation and easy real-time monitoring, relative independence from electromagnetic fields and rapid temperature changes, durability, fast response even at low concentrations, portability, low energy consumption and cost [8].

Several methods and techniques such as metal doping, application of high electric field, usage of nanosensing materials, micro-electromechanical system (MEMS) fabrication, decoration with noble metal nanostructures and ultraviolet (UV) illumination have been investigated to improve gas sensor performance [9–22].

A considerable number of works were devoted to enhancement of sensing properties of ZnO by UV illumination [9–13, 15–19, 21, 22]. During the sensing measurements, commercially available UV-LED light sources at three different wavelengths (365 nm, 395 nm or 405 nm) are used. On the other hand, there are a certain number of publications describing a sensing characteristic of a QCM coated with ZnO [8, 23–30]. One can note in this respect that we could not find any literature data concerning the sensing characteristics of a quartz resonator coated with a ZnO thin film under UV irradiation. To the best of our knowledge, this work is the first of its kind and would be considered a good basis for further study of the effects of the wavelength and power density on the gas-sensing properties of the quartz crystal microbalance sensor combined with a ZnO film.

In the present study, we report the elaboration and characterization of a zinc oxide thin film deposited onto QCM electrodes' surface using the radio-frequency (RF)-magnetron sputtering method for NH_3, H_2O_2 and ethanol gas sensors application at room temperature. The sensing properties of a zinc oxide-coated QCM sensor such as response/recovery time and sensitivity with and without UV illumination were investigated.

II. EXPERIMENT

ZnO thin films were deposited on both sides of the commercial quartz resonator HC49U by using the standard RF-magnetron sputtering method using a ZnO target in the argon atmosphere at the gas pressure of 0.1 Pa, under sputtering power of 100 W without substrate heating. The distance from the target to the substrate was 60 mm and the magnetic field strength

was 0.1 T. In order to remove any contamination, the target was presputtered for 1 min before deposition. The target was made of the pressed ZnO powder (99.99 % of purity). According to the ellipsometric measurements data, the thickness of the thin films was about 50 nm.

The *ex situ* ellipsometry measurements were performed with a serial null ellipsometer LEF-3 M in PCSA (polarizer–compensator–sample–analyzer) arrangement. The light source was He–Ne laser (λ = 632.8 nm).

The surface morphology of ZnO films was studied using a Solver P47-PRO atomic force microscope (AFM).

Gas sensing measurement of the QCM coated with ZnO thin films was conducted using a gas sensing system, as shown in Fig. 1. The resonant characteristics of the fabricated QCM device were examined using a quartz gauge of sprayed layer thickness KIT (SPE AKADEMPRYLAD LLC, Sumy, Ukraine) with the range of measured frequencies 4–10 MHz, a resolution of 1 Hz, and a measurement period of 1 s.

An UV-LED was used as the light source (λ_{max} = 395 nm; 2 mW/cm^2) for the UV illumination of the sensor. The distance between the UV-LED light source and the gas sensor was kept around 8 cm.

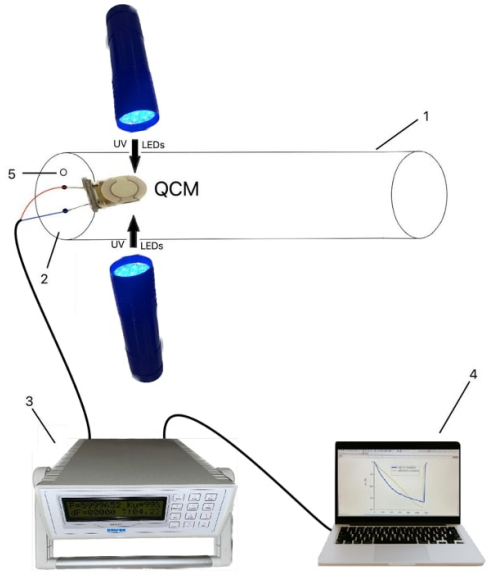

Fig. 1. Experimental setup used for the evaluation of the sensor performance: 1 — quartz tube; 2 — sealant; 3 — quartz gauge of sprayed layer thickness KIT (SPE AKADEMPRYLAD LLC, Sumy, Ukraine); 4 — computer; 5 — capillary for matter vapors delivery

III. RESULTS AND DISCUSSION

The surface morphology of ZnO thin films was monitored by AFM (Fig. 2). It was found that the obtained films were polycrystalline with an average grain size of about 36 nm and with a root mean square surface roughness of about 7 nm.

As is known, the sensitivity of the metal oxide to gas strongly depends on the grain size [31]. A smaller grain size increases the effective surface area of the film; that implies a tighter interaction of gas molecules with the film. As a result, sensitivity increases. A larger grain size means a smaller surface area of interaction with gas molecules. As a result, the sensitivity of the sensor decreases. Therefore, the grain size of the polycrystalline zinc oxide is reflected considerably in its gas sensing properties. It has been experimentally observed [32] that the gas sensitivity of ZnO sensors decreases with an increase in its mean grain size.

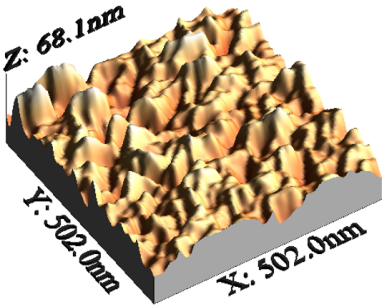

Fig. 2. 3D AFM image of ZnO thin film

The thickness of ZnO films in quartz crystal microbalance gas sensors varies in the range from 10 nm to 700 nm in different reports [8, 31–37]. The choice of the optimal film thickness of 50 nm was made on the basis of the analysis of the literature data [34, 38]. While the surface properties of the films are important in the application of QCM-based gas sensors, the effect of film thickness has not been thoroughly investigated yet. The thin films that could be prepared in a fast and cost-effective way are very promising for usage in the gas sensors.

According to the literature data, reactive oxygen ions, such as O_2^- , O^- and O^{2-}, are mainly chemisorbed in the dark on a surface of ZnO thin films with the aid of thermal energy [19, 39]. The reaction's kinetics can be described as follows [19, 39, 40]:

$$O_2(g) \rightleftarrows O_2(ads), \tag{1}$$

$$O_2(ads) + e^- \rightleftarrows O_2^-(ads), \tag{2}$$

$$O_2^-(ads) + e^- \rightleftarrows 2O^-(ads), \tag{3}$$

$$O^-(ads) + e^- \rightleftarrows O^{2-}(lat). \tag{4}$$

where the subscripts 'g', 'ads' and 'lat' mean gas, adsorbed and lattice, respectively.

UV light with the photon energy, not lower than the band gap of ZnO, stimulates carrier generation and consequently increases the density of free electron–hole

pairs in the ZnO films through the following reaction [15, 19, 40, 41]:

$$h\nu \rightarrow h^+ + e^-. \quad (5)$$

The photoinduced holes may interact with chemisorbed oxygen ions, causing the oxygen to be desorbed from the ZnO film surface according to the reaction [19]:

$$h^+ + O_2^-(\text{ads}) \rightarrow O_2(\text{g}). \quad (6)$$

Photogenerated oxygen ions are created due to the reaction of ambient oxygen molecules with the photoelectrons as follows [11, 15, 19]:

$$O_2 + e^-(h\nu) \rightarrow O_2^-(h\nu), \quad (7)$$

$$O_2 + e^-(h\nu) \rightarrow 2O^-(h\nu). \quad (8)$$

In contrast to the chemisorbed oxygen ions, which are strongly attached to the ZnO surface, these photogenerated oxygen ions are weakly bound and can be easily removed [19].

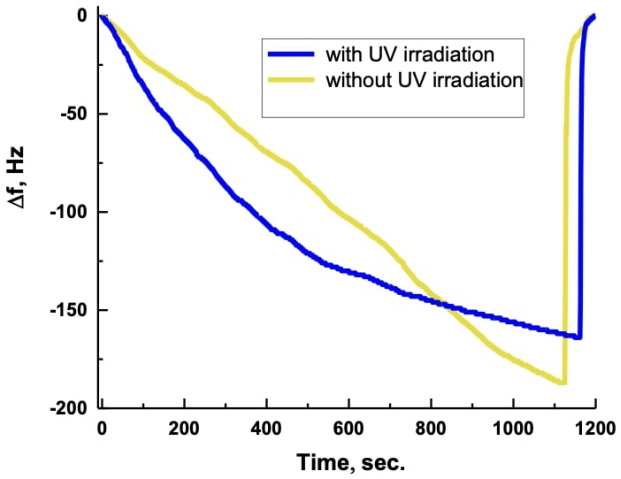

Fig. 3. The response curves of the fabricated sensor under the influence of the ethanol vapor at concentration of 2000 ppm at room temperature under UV light irradiation (blue line) and without UV light irradiation (yellow line)

Fig. 3 show the response transients of a ZnO coated QCM sensor to switching-on and off the ethanol vapor with 2000 ppm concentration at room temperature without and under UV illumination.

The ZnO-based sensor without UV illumination exhibits slightly higher response to C_2H_5OH in comparison to the case observed under the influence of UV illumination. But contrary to this, the response of the UV-illuminated ZnO sensor was more rapid with a more pronounced tendency toward gradual saturation. The recovery times of the sensor to 2000 ppm C_2H_5OH in the cases with and without UV light illumination was found to be about 8 s and 13 s, respectively.

The ethanol molecules will react rapidly with additional photogenerated oxygen ions on ZnO surface according to the following reaction [15, 19]:

$$C_2H_5OH + 3O_2^-(h\nu) \rightarrow 2CO_2 + 3H_2O + 3e^-. \quad (9)$$

The lower response of the sensor under UV light irradiation can be explained by a possible decrease in the ethanol vapor concentration in the quartz tube due to the photolysis reaction [42, 43]:

$$CH_3CH_2OH + h\nu \rightarrow CH_3CHO + 2H^+ + 2e^-. \quad (10)$$

The response transients of the ZnO coated QCM sensor to switching-on and off the ammonia vapor with 2000 ppm concentration at room temperature respectively without and under UV illumination are presented in (Fig. 4).

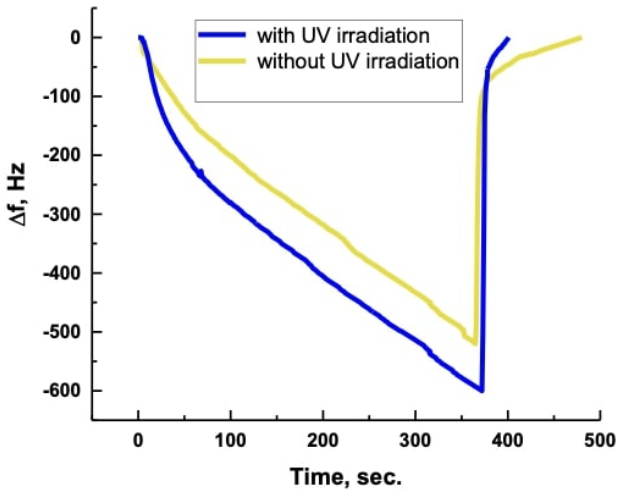

Fig. 4. The response curves of the fabricated sensor under ammonia vapor at concentration of 2000 ppm at room temperature under UV light irradiation (blue line) and without UV light irradiation (yellow line)

It is necessary to note that a ZnO-based sensor under the influence of UV illumination exhibits higher response compared to the case without illumination. The recovery times of the sensor to 2000 ppm NH_3 in these cases were found to be about 7 s and 29 s, respectively.

We detected the ammonia vapor in a medium-humidity environment, hence oxygen ions react with ammonia on the surface of the ZnO thin films and water ions participate in the reaction [44]. The reactions between oxygen ions and ammonia could be written as [40, 44]:

$$2NH_3 + 3O^-(h\nu) \rightarrow N_2 + 3H_2O + 3e^-, \quad (11)$$

$$2NH_3 + 5O^-(h\nu) \rightarrow 2NO + 3H_2O + 5e^-. \quad (12)$$

Physically sorbed H_2O also reacts with NH_3 to produce NH_4OH [24, 36]. Therefore, if NH_4OH is on

the surface of the ZnO thin films, it reacts with the photogenerated oxygen ions as [44]:

$$4NH_4OH + 7O_2^-(h\nu) \rightarrow 4NO_2 + 10H_2O + 15e^-. \quad (13)$$

In our case, ammonia photolysis did not occur, because photodissociation of NH3 takes place in the spectral range of light between 160 and 230 nm [45].

Fig. 5 presents the response transients of a ZnO coated QCM sensor to switching-on and off the hydrogen peroxide vapor with 2000 ppm concentration at room temperature without and under UV illumination.

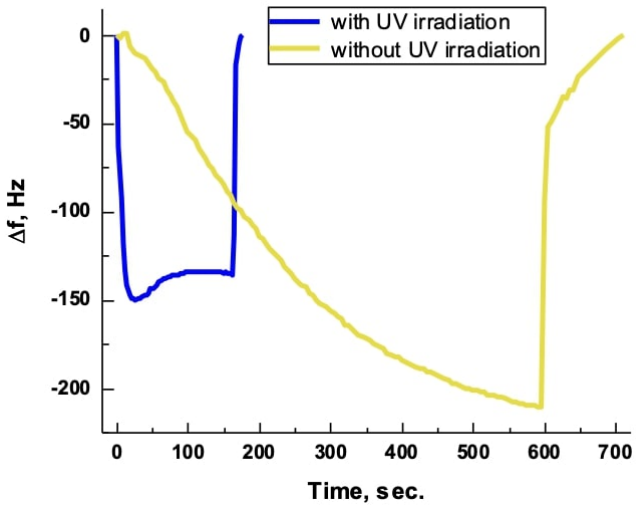

Fig. 5. The response curves of the fabricated sensor under hydrogen peroxide vapor at concentration of 2000 ppm at room temperature under UV light irradiation (blue line) and without UV light irradiation (yellow line)

In this case the ZnO-based sensor without UV illumination exhibits higher response compared to those under the influence of UV illumination. The response and recovery times of the sensor to 2000 ppm H_2O_2 in the case of UV light illumination were found to be about 12 s and 6 s, respectively. For comparison, these parameters for the sensor without UV light illumination were approximately 430 s and 59 s, respectively. The sensor with UV light illumination exhibits a more rapid response–recovery process to H_2O_2 vapor than in the case without UV light illumination.

Hydrogen peroxide molecules are split into the water and oxygen molecules on the ZnO thin films surface, then the oxygen molecules take electrons from the lattice, turning into oxygen ions [46–48]:

$$2H_2O_2 \rightarrow 2H_2O + O_2(g). \quad (14)$$

The hydrogen peroxide molecules also will react with additional photogenerated oxygen ions on the ZnO surface according to the following reaction [49, 50]:

$$2H_2O_2 + O_2^-(h\nu) \rightarrow 2H_2O + 2O_2(g) + 2e^-. \quad (15)$$

Taking into account that UV irradiation can photolyze H_2O_2 effectively, one can suggest that the visible light with the wavelength of 405 nm also would photolyze H_2O_2 [51, 52]:

$$2H_2O_2 + h\nu \rightarrow 2H_2O + O_2(g). \quad (16)$$

Photolysis of hydrogen peroxide can be the reason for a lower response of the sensor under the influence of UV light irradiation.

The additional experiments were carried out when the UV-LED illumination was applied only during the recovery period of a QCM sensor covered with a ZnO film after detection of ethanol, ammonia or hydrogen peroxide vapor. In this case, a reduction in the recovery time of the sensor during the detection of ethanol vapor, ammonia or hydrogen peroxide by 2.6, 2, and 1.8 times, respectively, was revealed (Fig. 6)

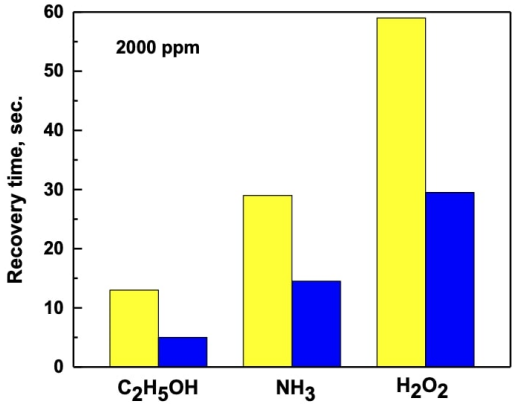

Fig. 6. Illustration of the effect of UV LED illumination on the recovery time of a QCM sensor coated with a ZnO film after detection of different vapors

Therefore, the obtained experimental data and calculated characteristics prove that UV-LED illumination of the QCM sensor covered with a ZnO film leads to a significant reduction in its recovery time at detection of ethanol, ammonia or hydrogen peroxide vapors. At the same time, during the operation of the illuminated UV-LED QCM sensor covered with a ZnO film, it is necessary to take into account the possible photolysis of the analyzed substances.

IV. CONCLUSIONS

A quartz crystal resonant gas sensor with ZnO thin films was successfully fabricated and tested. The obtained results showed that the recovery time of the sensor for detection of ethanol, ammonia or hydrogen peroxide vapors is considerably reduced due to UV-LED irradiation. UV-LED illumination opens the possibility for improvement of the commercial sensors, in particular, sensors of nitrate and chlorate compounds and hydrogen peroxide – components of explosive devices.

V. ACKNOWLEDGMENTS

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ВПЛИВ УЛЬТРАФІОЛЕТОВОГО ВИПРОМІНЮВАННЯ НА ГАЗОЧУТЛИВІ ВЛАСТИВОСТІ КВАРЦОВО-КРИСТАЛІЧНИХ СЕНСОРІВ МІКРОБАЛАНСУ ПОЄДНАНИХ ІЗ ПЛІВКОЮ ZnO

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Тонкі плівки цинк оксиду осаджено на обидва електроди комерційного кварцового резонатора HC49U високочастотним магнетронним розпиленням для детектування пари NH_3, H_2O_2 та C_2H_5OH за кімнатної температури. Згідно з даними еліпсометричних досліджень товщина тонких плівок ZnO приблизно дорівнювала 50 нм. Для моніторинґу в морфології поверхні тонких плівок ZnO використано атомно-силову мікроскопію. Установлено, що отримані плівки були полікристалічними із середнім розміром кристалітів приблизно 36 нм та зі середньоквадратичною шорсткістю поверхні приблизно 7 нм.

Зміну частоти виготовленого кварцового резонатора, вкритого тонкими плівками цинк оксиду, за умови наявності або відсутності аналізованих речовин реєстрували кварцовим вимірювачем товщини напиленого шару КІТ (ТОВ "НВП Академприлад", м. Суми, Україна), який має діапазон вимірюваних частот 4–10 МГц, роздільну здатність 1 Гц і період вимірювання 1 с.

Порівняно з іншими типами сенсорів перевагами газових кварцових сенсорів мікробалансу є: висока чутливість; здатність працювати за кімнатної температури; проста технологічна реалізація та легкий моніторинґ у режимі реального часу; відносна незалежність від електромагнітних полів та швидких змін температури; довговічність; швидка реакція навіть за низьких температур та концентрації; транспортабельність; низьке енерґоспоживання та вартість.

Уперше досліджено вплив освітлення ультрафіолетовим світлодіодом (з максимумом смуги випромінювання світла за $\lambda_{max} = 395$ нм і питомою потужністю випромінювання 2 мВт/см^2) на основні характеристики вкритого ZnO кварцового сенсора мікробалансу, як-от: час відгуку/відновлення та чутливість. Отримані експериментальні дані та розрахункові характеристики свідчать, що ультрафіолетове опромінення вкритого плівкою ZnO кварцового сенсора мікробалансу приводить до значного скорочення часу його відновлення під час детектування пари етанолу, аміаку або пероксиду водню. Водночас за постійного освітлення ультрафіолетовим світлом такого сенсора необхідно враховувати можливість протікання реакцій фотолізу аналізованих речовин.

Виготовлений кварцово-кристалічний сенсор мікробалансу з тонкоплівковим покриттям ZnO за умови опромінення ультрафіолетовим світлом продемонстрував багатонадійний потенціал в удосконаленні давачів нітратних сполук та пероксиду водню — складових вибухових пристроїв.

Ключові слова: освітлення ультрафіолетовим світлодіодом, газовий сенсор, тонка плівка, кварцово-кристалічний сенсор мікробалансу.