PHYSICS AND CHEMISTRY OF SOLID STATE
V. 23, No. 2 (2022) pp. 361-367
Section: Physics

DOI: 10.15330/pcss.23.2.361-367

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ФІЗИКАІХІМІЯТВЕРДОГОТІЛА
Т. 23, № 2 (2022) С. 361-367
Фізико-математичні науки

UDC: 538.911 ISSN 1729-4428

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Formation of CdO/CdS/textured-ZnO/ZnO heterostructures by chemical deposition

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CdO/CdS/textured-ZnO/ZnO heterostructure was synthesized using a combination of electrochemical etching and chemical deposition methods. Electrochemical etching was used to form a textured ZnO layer. Chemical deposition of a solution containing $CdCl_2$ was performed to prepare a CdS film with CdO nanoparticles on the surface. The obtained nanocomposite was characterized using SEM, EDX and Raman methods for structural, morphological and component studies. SEM images and Raman scattering showed the existence of cubic phase of CdO nanocrystals. The SEM study revealed the dispersion of 50-200 nm agglomerated nanostructures on the surface of CdS film.

Keywords: electrochemical etching, chemical deposition, nanostructures, Raman scattering, heterostructure.

Received 2 May 2022; Accepted 23 June 2022.

Introduction

Metal oxides have been the subject of many studies due to their excellent photocatalytic properties. TiO_2 [1,2], NiO [3, 4], Ga_2O_3 [5, 6], MgO [7, 8], CdO [9, 10], etc. semiconductors are widely studied. These semiconductors are usually characterized by long diffusion length and ease of synthesis. Numerous studies have been devoted to ZnO material due to its wide range of applications, from optoelectronics to cosmetology [11, 12]. As a semiconductor, ZnO is used in piezo electronics, sensor devices, and optoelectronics [13-14]. However, a significant drawback for certain applications of this semiconductor should be noted, namely the wide forbidden area (E_g=3.2 eV) [15]. This property may limit the absorption of the visible light spectrum. Narrow-gap semiconductors are deposited on the ZnO surface to generate electron-hole pairs in the visible region of the spectrum. Thus, a c-Si/ZnO heterostructure for ultra-thin solar cells has been reported [16]. Ag_3VO_4/ZnO nanocrystal structure is proposed as a visible light photocatalyst [17]. The synthesis of ultrathin CdS layer on an array of ZnO nanorods using pulsed electrodeposition has been reported to improve photo-charging transportation under the visible spectrum [18]. In [19], a CdS layer was also applied to ZnO nanotube arrays to increase photocatalytic activity.

Cadmium sulphide (CdS) is a semiconductor, which is widely used in optical electronics and continues to be the subject of many studies due to its properties. In particular, CdS has a direct forbidden area of 2.42 eV. Another interesting property of cadmium sulphide is its ability to nanostructure. The synthesis of nanoneedles, nanorods, nanowires, etc. has been reported [20, 21]. However, the growth of CdS films on ZnO substrates has many problems and limitations. For example, the mismatch of crystal lattice parameters leads to film cracking and excessive stresses. In addition, charge transfer can only be effective at the active interface, while the recombination of electron-hole pairs increases as the film thickness increases. This fundamentally affects the photoactivity of the heterostructure [19]. To eliminate these problems, we report a simple method of texturing the ZnO substrate to create a buffer layer between the main substrate and the CdS structures in this study. Such buffer substrates are often porous layers formed on the surface of the main substrate [22]. For example, we reported the growth of indium nitride on an

indium phosphide surface with a por-InP buffer layer [23]. The porous layer was shown to be a "soft" substrate, allowing stresses due to crystal lattice mismatch to be minimized. Moreover, porous layers on monocrystalline semiconductors increase the roughness and texture of the surface, which provides better adhesion of the synthesized nanostructures to the substrate material [24]. In addition, we modify the CdS surface by electrochemical synthesis of CdO nanocrystals to improve the photo-electrochemical properties and provide passivation of the surface of the formed structure.

I. Samples and experimental methods

The experiment was conducted in two stages.

The first stage is the formation of a textured layer on a monocrystalline ZnO surface. The purpose of this stage is to relieve excessive stresses on the substrate surface and form textures and pores that will ensure the adherence of nanoparticles to the crystal surface. The textured ZnO layers were formed by electrochemical etching in electrolyte solution $HCl:H_2O:C_2H_5OH=2:1:1$. Etching was performed in a standard three-electrode cell at a constant voltage U = 5V for 10 minutes. During electrochemical treatment, the samples were illuminated with a 250W Osrm XBO xenon lamp at a distance of 10 cm from the semiconductor surface.

To prepare CdS nanoparticles, cadmium chloride solution (0.1 M $CdCl_2$) was used, which was dissolved in 100 ml of distilled water (DI) and thiocarbamide (0,1 M CH_4N_2S) and ammonia (5M NH_3) were added. The solution was mixed with a magnetic stirrer for 20 minutes and heated to a temperature of 80ºC. Textured ZnO samples were immersed in the prepared solution and maintained for 5 hours. After that, the samples were removed from the solution and left in the open air for 3 months.

The morphology of the nanostructures was characterized using an SEO-SEM Inspect S50-B scanning electron microscope. The SEM was also equipped with an EDX spectrometer used for elemental analysis (AZtecOne with X-MaxN20 detector). Raman spectroscopy was performed at room temperature using the RENISHAW inVia Reflex system with 532 nm generation wavelength at 0.5% intensity, 2400 nm lattice, 100-1000 cm^{-1} range. The measurement time was 10 s and 5 accumulations were performed.

II. Results

2.1 SEM analysis

After the electrochemical treatment of the ZnO samples, their surface became textured. Fig. 1 demonstrates the elements of the surface of zinc oxide samples. From the figure, we can see that the surface has become rough, and sliding dislocations can be observed (Fig. 1 a, b). We can also see the formation of etching pits on the surface, namely two types of pores. We can see mall pores in Figure 1(a). The diameter of such pores is (50-150) nm. Such pores form on the crystal surface last, have a spontaneous character of formation and are not related to surface defects and defects in the crystal lattice of the original semiconductor [25]. The second type of pores (Figure 1c, d) is massive star-shaped etching pits, the diameter of which is in the range of (5–10) microns. The occurrence of these pores is associated with dislocations reaching the surface, resulting in crater formations. The surface is also characterized by dots up to 1 μm in diameter (Figure 1b, d). Most likely, the dots are the result of the sedimentation of reaction products that occur during the electrochemical dissolution of the crystal.

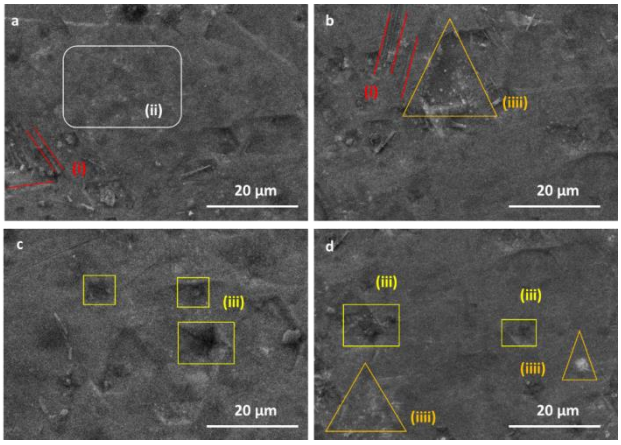

Fig. 1. Textured ZnO surface after electrochemical treatment: (i) sliding dislocation outcrop, (ii) fine micropores, (iii) star-shaped pores, (iiii) oxide inclusions.

In general, semiconductors of the A^2B^6 group, in contrast to semiconductors A^3B^5, show poor ability to structure during electrochemical treatment [26]. For example, the formation of a wide class of nanostructures on the surface of indium phosphide, gallium arsenide, and gallium phosphide is observed in a wide range of etching regimes [27, 28]. The surfaces of ZnO, ZnSe, ZnTe and others are quite stable for many electrolytes [29, 30]. On the other hand, A^2B^6 semiconductors have good chemical inertness concerning air and moisture. Their surface is almost non-oxidising and does not require surface passivation [31]. This fact gives such semiconductors a significant advantage over others in many aspects.

The purpose of this stage of the experiment was not to form ordered structures on the ZnO surface, it was only necessary to texture the surface and etch out lattice defects to reduce excessive stresses. We can state that this purpose was achieved.

Figure 2 shows the morphology of the sample after the chemical deposition.

We can see that the surface morphology is significantly changed. For example, etch pits are no longer observed on the surface, there are only thin long stripes, ranging in size from 75 to 500 nm in cross-section. In addition, spherical crystallites appeared on the surface. Due to the specularity of the sample, the microphotographic quality is rather poor. However, three characteristic spherical sizes can be observed, namely (i) 80 nm, (ii) 500 nm, and (iii) 1 μm.

Thus, we can conclude that a dense film has formed on the surface of textured ZnO. It lines the pores and pits

of etching and is tightly bound to the substrate material. The spheres formed on the surface are another phase; they were formed after the film formation process was completed. It is logical to assume that these are oxide compounds.

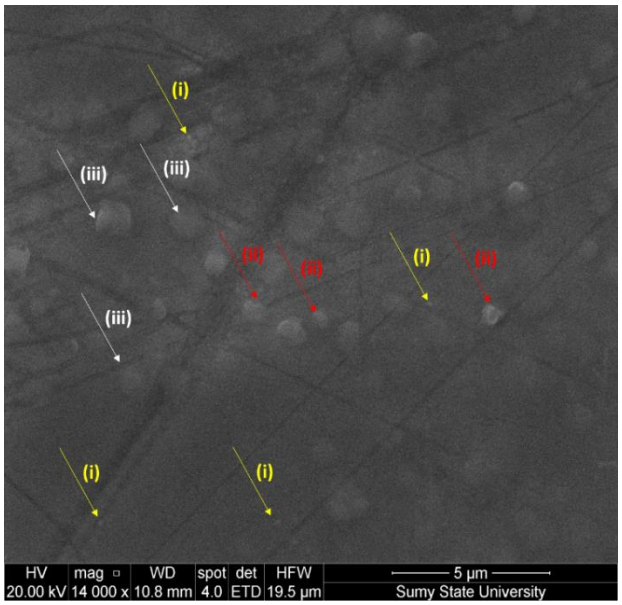

Fig. 2. Morphology of film, microparticles and nanoparticles on ZnO surface.

2.2 EDX analysis

Analysis of the elemental composition of the resulting structure (Figure 3) shows the presence of oxygen, zinc, cadmium, sulphur and carbon on the surface. The presence of carbon may indicate that complex compounds of reaction products have been formed on the surface of the structure. Most likely, a passive film does not contribute to the overall surface properties of the material. It should be noted that reflexes from other elements are strong enough, i.e. they shine through the passive film. The emergence and contribution of carbon needs further study.

In percentage terms, we can observe that there is almost twice as much oxygen on the surface as zinc (Table 1). From this, we can conclude that there are other compounds other than ZnO containing oxygen on the surface. Inclusions of cadmium oxide could be the most likely source of oxygen. The appearance of this compound can be due to the oxidation of surface-bound cadmium atoms with oxygen ions in the electrolyte. Oxygen can also attach to surface cadmium atoms, which are characterized by the presence of broken bonds and chemical activity.

Table 1.

Elemental composition of the sample surface obtained using the EDX method.

Element	Line type	Atom., %
C	K-series	21.82
O	L-series	25.12
S	K-series	20.69
Zn	K-series	3.23
Cd	L-series	29.14

2.3 Raman spectroscopy analysis

The study of Raman scattering showed that the Raman spectrum has five peaks: 112, 210, 254, 300, and 602 cm^{-1} (fig. 4). 300 and 602 cm^{-1} intense peaks caused by LO main phonon mode and 2LO CdS, the first supertonic mode, respectively. This agrees well with previous studies [32]. In addition, the study [33] reports that the 300 cm^{-1} peak is due to the presence of nanometer-sized particles. This is consistent with the scanning electron microscopy results presented in Figure 2.

In nanostructured semiconductors, the exciton-phonon bond strength is estimated as the ratio of the overtone intensity to the main one. For our structure, this coefficient is equal to I_{2Lo}/I_{Lo} = 1.65. Compared to other similar studies [34], the value of this ratio is quite high, which indicates a strong exciton-LO phonon bond in the synthesized structure.

We can observe that the first-order Raman light scattering line LO is not only extended but also asymmetric towards a higher frequency compared to the typical CdS volume spectrum (305 cm^{-1}). This also indicates the presence of particles on the surface that have a significant variation in size. A sharp rise in the spectrum at 605 cm^{-1} or more with no visible peaks could

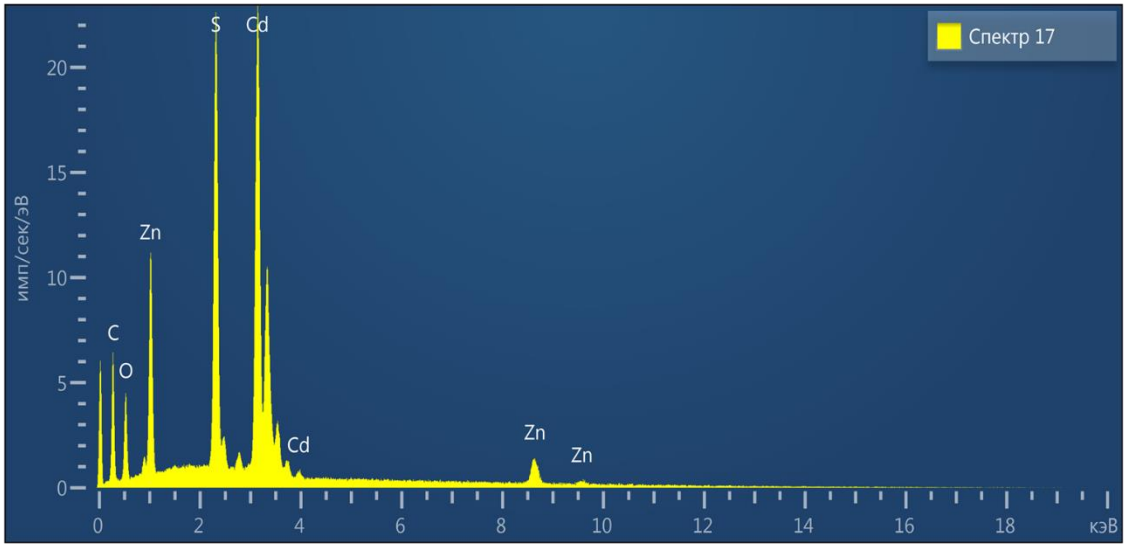

Fig. 3. ZnO surface EDX spectrum after chemical deposition.

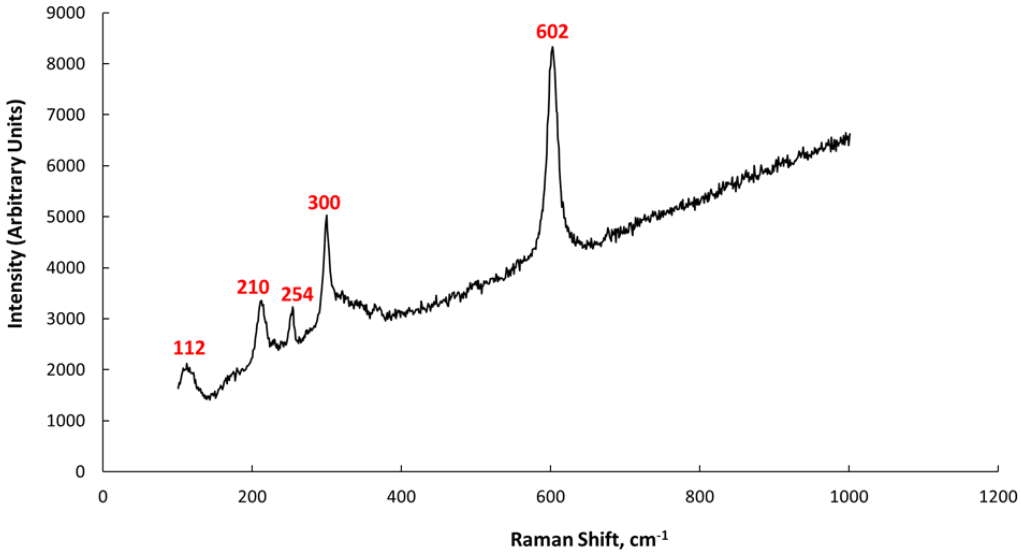

Fig. 4. Raman spectra of nanostructures, which were synthesized on ZnO surface.

indicate multiphoton scattering. The 254 cm^{-1} peak is typical for a cubic CdO phase [35]. Thus, we can state that the spherical nanocrystals on the resulting structure surface are cadmium oxide. This is consistent with the previously made assumption based on the results of chemical analysis obtained by the EDX method. The crystallinity of this formation is also indicated by the fact that the peak is 254 cm^{-1} is quite narrow. In addition, the 210 cm^{-1} peak also appears due to the presence of CdO nanoparticles. The shift to the low-frequency part of the spectrum compared to the typical peak (216 cm^{-1}) also indicates the presence of nanometer-sized crystallites [36]. 112 cm^{-1} mode is most likely related to the nanopore textured ZnO surface, which is the substrate for the synthesized CdO/CdS structure. That is, we have a complex CdO/CdS/textured-ZnO/ZnO structure (Fig. 5). These heterostructures can be used in optoelectronic devices. Subsequent research should focus on the improvement of heterostructure synthesis technology and direct evaluation of their photocatalytic properties.

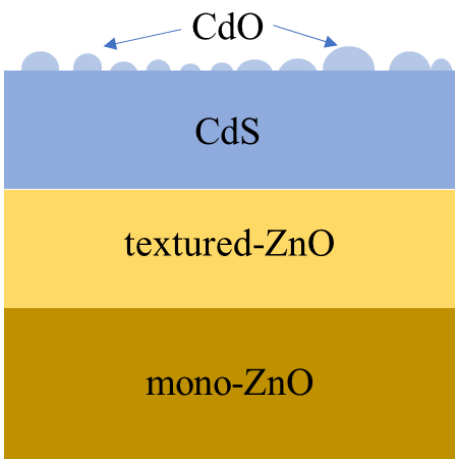

Fig. 5. Schematic representation of CDO/CdS/ textured-ZnO /ZnO heterostructure.

Conclusions

Substrates made of textured ZnO were prepared by electrochemical etching. The CdS film with CdO nanocrystallites was prepared by chemical deposition. Nanocrystallites are spherical and have a cubic crystal lattice. The shift of Raman spectra to the low-frequency region indicates the presence of nanometer-sized particles. The SEM analysis of the surface micromorphology also shows the nanometer-sized particles with a diameter of 80 nm. Texturing of ZnO before chemical deposition of the substrate allowed the formation of a high-quality CdO/CdS/textured-ZnO/ZnO crystal heterostructure.

Acknowledgment

The study was supported by the Ministry of Education and Science of Ukraine, namely:

- the state budget research project No. 0122U000129 The search for optimal conditions for nanostructures synthesis on the surface of A3B5, A2B6 semiconductors and silicon for photonics and solar energy;

- project No. 0121U10942 Theoretical and methodological bases of the system fundamentalization of the future nanomaterials experts training for productive professional activity.

Ya. Sychikova thanks Goethe-Institut for supporting the House of Europe graduate emergency scholarship programme.

We also thank the Armed Forces of Ukraine for the safety to carry out this work. This work was only possible thanks to the resilience and courage of the Ukrainian Army.

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Формування гетероструктур CdO/CdS/textured-ZnO/ZnO методом хімічного осадження

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Гетероструктуру CdO/CdS/textured-ZnO/ZnO синтезували з використанням комбінації методів електрохімічного травлення та хімічного осадження. Електрохімічне травлення було застосовано для формування текстурованого шару ZnO. Хімічне осадження розчину, що містив $CdCl_2$ здійснювали для приготування плівки CdS з наночастиками CdO на поверхні. Отриманий нанокомпозит був охарактеризований за допомогою SEM, EDX та Raman методів для структурних, морфологічних та компонентних досліджень. Зображення SEM та спектри комбінаційного розсіювання світла показали існування кубічної фази нанокристалітів CdO. Дослідження SEM виявило дисперсію агломерованих наноструктур розміром 50–200 нм на поверхні плівки CdS.

Ключові слова: електрохімічне травлення, хімічне осадження, наноструктури, комбінаційне розсіювання світла, гетероструктура.