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Color-Tunable Photoemission in Alq$_3$ – Porous Silicon Hybrid Structures

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Abstract. The possibility of tuning the photoemission color of hybrid structures obtained by thermal deposition of a tris(8-hydroxyquinoline) aluminum thin film on the surface of photoluminescent porous silicon was studied in the present work. It is established that the deposited film is sufficiently transparent in the visible spectral range but partially absorbs UV radiation. The photoluminescence excitation and emission spectra of the obtained hybrid structures were investigated in the ranges of 220–400 and 450–800 nm, respectively. The possibility of changing the ratio of the intensities of the green and red photoemission bands in the hybrid structure by controlling the thickness of the metal-organic film and by varying the photoluminescence excitation energy has been demonstrated.

Keywords: porous silicon, tris(8-hydroxyquinoline) aluminum, hybrid structure, photoluminescence, light transmittance
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1. Introduction

Current trends in the development of optoelectronics are closely related to nanostructured materials with attractive properties arising from quantum-size effects. In particular, the intense visible photoluminescence of porous silicon (PS) nanostructures can be used to create a new generation of optoelectronic devices, extending the functionality of silicon technology [1-4]. The possibility of tuning the properties of silicon nanocrystals to meet specific needs by changing their size or shell is also interesting [5-7]. In addition, such combining can facilitate the integration of materials of different natures into modern microelectronic technology.

The branched pore system not only increases the light-absorbing surface area but also makes the PS an ideal candidate as a host matrix for the deposition of various materials, including conjugated polymers for the formation of photovoltaic structures [8-10]. Hybrid organic-inorganic structures may also have high potential for application in photoemission devices [11]. In particular, small-molecule metal-organic compounds, such as tris(8-hydroxyquinoline) aluminum (Alq$_3$), are promising for the development and production of organic light-emitting diodes and transistors due to their uniform emission over large areas, adjustable emission wavelength, and low manufacturing cost [12, 13]. Alq$_3$ is also characterized by high quantum efficiency of photoluminescence (PL) [14-16]. The wavelength of green photoemission can be tuned by attaching donor or acceptor substituents to the quinoline ligand, which changes the band gap of Alq_3, or by introducing ZnO quantum dots into the metal-organic compound [17,18]. Furthermore, Alq_3 hybridized with silver or aurum nanoparticles demonstrates increased PL efficiency at resonant wavelengths due to plasmon resonance [19, 20].

Alq_3 deposition onto the PS layer can, on the one hand, improve the surface passivation of luminescent silicon nanocrystals, as observed with polyaniline deposition [21], and, on the other hand, provide multicolor PL of the formed hybrid structure, similar to that of Alq_3–ZnO nanocomposite photoemission [18]. Therefore, the purpose of the work was to study the photoluminescent properties of the Alq_3–PS structures. Particular attention was focused on the possibility of controlling the hybrid structure PL spectrum.

2. Experiment

Nanostructured PS layers on silicon substrates, obtained as a result of electrochemical etching of polished wafers of electronic conductivity type with a thickness of 400 μm in an ethanolic solution of hydrofluoric acid, were used as the basis of the Alq_3–PS luminescent structures. The anodic current density was 30 mA/cm^2 throughout the etching process, which lasted 10 min. The working surface of the silicon wafer was irradiated with a 500 W incandescent lamp during etching to generate the positive charge carriers required for PS formation. After washing in distilled water and drying in air, the resulting structures were used as substrates for Alq_3 deposition.

An Alq_3 powder with a purity of 99.995% produced by Sigma-Aldrich was used to form a thin film on the surface of the PS nanostructures. The metal-organic film was formed by thermal evaporation of Alq_3 powder in a vacuum and subsequent deposition onto the surface of the porous layer. The film thickness was determined during the deposition process using a quartz thickness gauge and controlled by the duration of thermal evaporation. As a result, the Alq_3–PS hybrid structures with metal-organic film thicknesses of about 50, 100, and 150 nm were obtained. Additionally, the Alq_3 films with the same thicknesses were deposited on glass substrates to study their UV-Vis transmittance spectra.

The obtained structures were characterized using atomic force microscopy (AFM). The photoluminescent properties of the Alq_3–PS structures were investigated using a fluorometer Solar SM2203. In particular, the photoemission spectra of the experimental samples excited by UV radiation were measured in the 450–800 nm range. The PL excitation spectra were examined in the range of 220–400 nm for luminescence band maxima. The UV-Vis transmittance spectra of the ALq_3 thin films were measured in the range of 220–800 nm. All measurements were carried out in air at room temperature.

3. Results and discussion

The surface topography of the Alq_3 film and Alq_3–PS hybrid structure was studied using AFM in the tapping mode. Fig. 1 shows the two-dimensional micrograph of a 1×1 μm^2 area of the experimental samples. AFM studies did not reveal a regular structure of the Alq_3 film on the glass substrate, which may indicate the amorphous nature of the obtained film (see Fig. 1a). The Alq_3 film, after thermal deposition on the PS surface, repeated the topology of the porous layer. As a result, objects with sizes ranging from several tens to hundreds of nanometers were observed (see Fig. 1b). We assume that these are remnants of pore walls covered with the metal-organic film.

The PL spectra of the main components of the Alq_3–PS hybrid structure were investigated to identify features of its photoemission properties through comparative analysis. The measured visible luminescence spectra of the initial PS and Alq_3 films on a glass substrate, excited by UV radiation, are shown in Fig. 2. The PS and Alq$_3$ demonstrate intense red and green PL bands, respectively, and their spectral positions correspond to those reported by other researchers [14, 22]. It should be noted that the intensity of the Alq$_3$ PL depends on the film thickness. In particular, an increase in the thickness from 50 to 150 nm causes an increase in the photoemission intensity by several times.

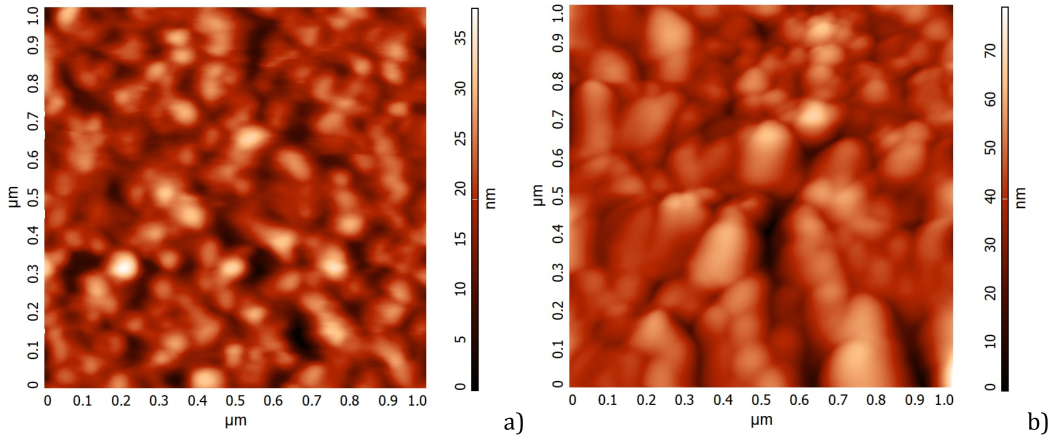

Fig. 1. Two-dimensional AFM micrographs of the Alq$_3$ film on the glass substrate (a) and Alq$_3$–PS hybrid structure (b).

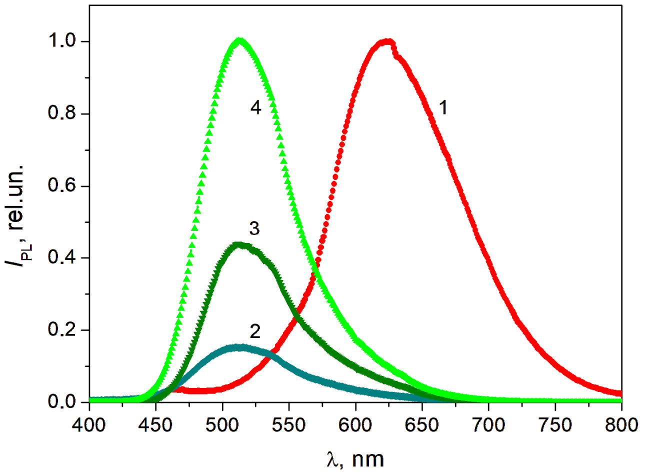

Fig. 2. Normalized PL spectra of the PS (1) and Alq$_3$ films (2, 3, 4) on a glass substrate. Curves 2, 3, and 4 refer to Alq$_3$ films with thicknesses of 50 nm, 100 nm, and 150 nm, respectively.

The experimental samples of the Alq$_3$–PS hybrid structures exhibit a more complex photoemission spectrum in the visible range when excited by UV radiation (Fig. 3). In particular, the PL spectra are characterized by two visible bands of different natures. A broad band with a maximum in the region of 600–650 nm is apparently associated with quantum confinement in the PS nanostructures. It is formed due to radiative recombination of photogenerated electron-hole pairs in silicon nanocrystals with the size dispersion [23]. As a result, the PL band of the PS is broad, and its maximum can be at different wavelengths of the visible spectrum, depending on the conditions of the porous layer formation and the degree of passivation of the surface of silicon nanocrystals [21, 22]. In addition, passivation of silicon dangling bonds reduces the fraction of non-radiative recombination of non-equilibrium charge carriers, increasing the PL quantum efficiency of the PS nanostructures.

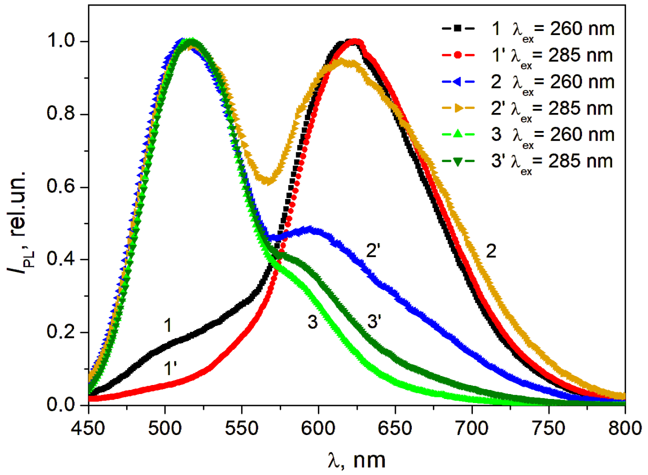

Fig. 3. Normalized PL spectra of the Alq$_3$–PS hybrid structures with the Alq$_3$ film thicknesses of 50 nm (curves 1 and 1'), 100 nm (curves 2 and 2') and 150 nm (curves 3 and 3') excited by UV radiation with wavelengths of λ_{ex} = 260 nm (1, 2, 3) and 285 nm (1', 2', 3').

Along with the red PL band, a green band with a maximum at about 515 nm, which is associated with the metal-organic compound Alq$_3$, is observed. In general, the optical and luminescent properties of Alq$_3$ depend on its structural characteristics in the solid state [24]. In addition to the amorphous state of the metal-organic compound, which is usually characteristic of thermally deposited films, several different polymorphic crystalline phases of Alq$_3$ are distinguished. The polymorphism of Alq$_3$ is due to the peculiarities of the chemical structure of Al(C$_9$H$_6$NO)$_3$ (see the inset of Fig. 4). The compound Alq$_3$ contains three ligands that surround the central aluminum ion (Al^{3+}) and can have different geometric arrangements, which affect the molecular density of the packing. Although aluminum itself does not participate in the emission of light directly (the optical transition responsible for the PL of Alq$_3$ is centered on the organic ligand), its role is to provide a rigid structure that locks the ligands in a specific position. As a result, a distinction is made between meridional and facial isomeric forms of the molecule [25]. The dominant isomeric form of the material is important, as the photoemission characteristics of the meridional and facial isomers are somewhat different [24, 25].

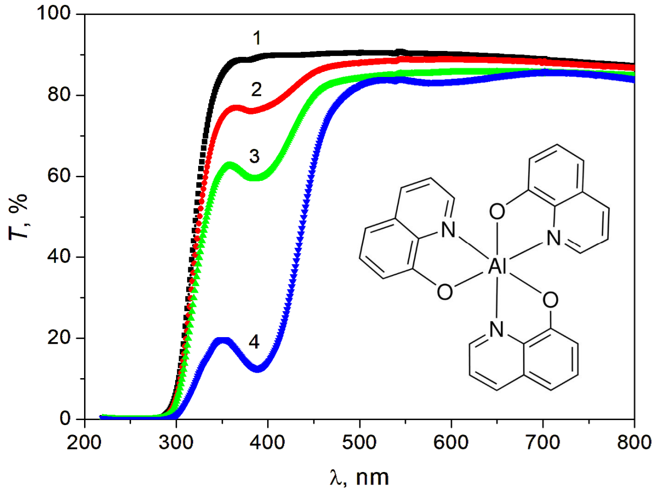

Fig. 4. UV-Vis transmittance spectra of the glass substrate (1) and Alq$_3$ film with the thicknesses of 50 nm (2), 100 nm (3), and 150 nm (4) on the glass substrate. Inset: the chemical structure of Alq$_3$.

It should be noted that despite the high thermal stability of Alq_3, its long-time UV irradiation (for tens and hundreds of hours) causes a decrease in the PL efficiency due to photooxidation of the metal-organic compound in the air atmosphere [26]. As a possible solution to overcome these problems, the use of hybrid (metal-organic/inorganic) composites has been proposed [18, 27]. On the other hand, replacing unstable surface complexes Si-H_x (x = 1, 2, 3) on the PS surface [21] with a more stable Alq_3 compound can provide higher stability of the photoluminescent properties of the PS nanostructures.

The ratio between the intensities of the green and red photoemission bands was different for various experimental samples of the Alq_3–PS hybrid structures. In particular, increasing the thickness of the Alq_3 film causes an increase in the intensity of photoemission with a maximum in the range of 500–530 nm and a decrease in the PL band intensity in the 600–650 nm range. The observed transformation of the PL spectra of the Alq_3–PS hybrid structures with different Alq_3 film thicknesses may be due to partial absorption by the film of both the radiation exciting the luminescence of the PS nanocrystals and the red band of the photoemission itself. On the other hand, a larger thickness of the metal-organic film provides a larger number of radiative recombination centers associated with the quinoline ligands, and therefore a higher intensity of the green band of the PL.

To obtain additional information about the effect of the thickness of the Alq_3 film on the PL of hybrid structures, the UV-Vis transmittance spectra of films with a thickness of 50, 100, and 150 nm deposited on glass substrates were investigated. For comparison, the transmittance spectrum of the glass substrate was also measured. As can be seen in Fig. 4, the Alq_3 films are sufficiently transparent in the visible spectral range and can serve as a "window" for the PS luminescence. However, they are not transparent in the UV range. Moreover, the intensity of UV radiation absorption, namely in the absorption band at about 390 nm, increases significantly with increasing Alq_3 film thickness. Given that this absorption band is attributed to the π–π^* electronic transition from the highest occupied molecular orbital (HOMO), which lies predominantly on the phenoxide ring, to the lowest unoccupied molecular orbital (LUMO) that is located on the pyridyl ring of the ligand [16, 28], a larger Alq_3 film thickness causes an enhancement of the green emission band. In addition, the Alq_3 film partially blocks the radiation that excites the PS PL.

It should be noted that the intensity of the PL bands of the Alq_3–PS structures depends on the energy of excitation quanta, as shown in Fig. 3. This fact can be used to tune the color of photoemission by changing the PL excitation wavelength. However, the efficiency of controlling the PL spectrum of the Al_3–PS hybrid structure depends on the thickness of the Al_3 film. Its tiny thickness does not provide sufficient intensity of the green PL band compared to the red band of the PS photoemission. Conversely, an excessively thick metal-organic film almost completely quenches the light emission from the PS, likely due to the strong absorption of the PL excitation quanta.

To confirm this assumption, the PL excitation spectra of the Alq_3–PS hybrid structure were investigated for wavelengths corresponding to the maxima of the photoemission bands and compared with the PS and Alq_3 film PL excitation spectra. As shown in Fig. 5, the PL excitation spectra of the Alq_3 film and the Alq_3–PS structure are similar for λ_{PL} = 515 nm. In addition to a broad band in the range of 360-400 nm, which correlates with the absorption band in the UV-Vis transmittance spectra of the Alq_3 films, these PL excitation spectra have an intense band with a maximum at about 260 nm.

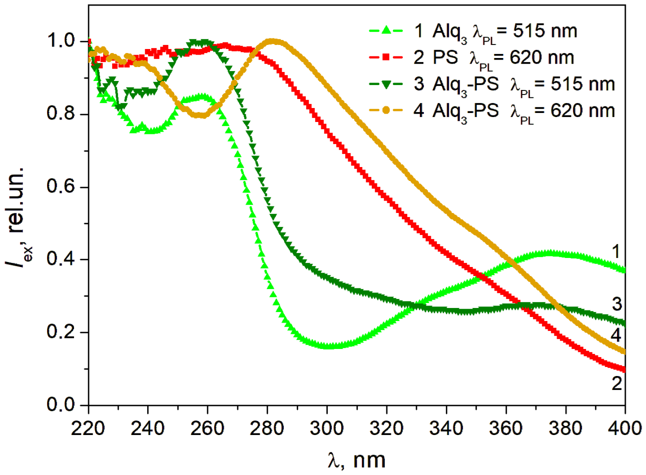

Fig. 5. Normalized PL excitation spectra of the Alq$_3$ film (1), PS (2), and Alq$_3$–PS hybrid structure (3, 4) for λ_{PL} = 515 nm (1, 3) and 620 nm (2, 4).

The PL excitation spectra of the PS and Alq$_3$–PS hybrid structure for λ_{PL} = 620 nm are characterized by a broad band in the UV range, the intensity of which decreases with increasing wavelength from 290 to 400 nm. The slight broadening of the excitation band to the lower energy region observed for red photoemission in the Alq$_3$–PS structure may be due to the passivation of the luminescent PS nanocrystal surface by the metal-organic film. In addition, a decrease in the intensity of excitation of the red PL band of the hybrid structure at about 260 nm was detected, which is likely due to an increase in the absorption of radiation by the Alq$_3$ film in this region. As a result, the color of the photoemission in the Alq$_3$–PS structures can be tuned within the range of 515–620 nm by changing the wavelength of the PL-exciting radiation in the range of 260–290 nm.

4. Conclusions

The Alq$_3$–PS hybrid structures were obtained by thermal deposition of a thin Alq$_3$ film onto the surface of the photoluminescent PS formed on a silicon substrate via electrochemical etching. The created structures are characterized by two intense photoemission bands with maxima near 515 and 620 nm when excited with UV light. The green PL band is associated with the Alq$_3$ film, and the red band is related to the PS. Based on the comparative analysis of the UV-Vis transmittance spectra of Alq$_3$ films with different thicknesses, it was established that the deposited films are transparent to the PS luminescent radiation but partially absorb UV radiation. The possibility of changing the ratio between the intensities of the green and red PL bands of the hybrid structure by controlling the thickness of the Alq$_3$ film from 50 nm to 150 nm has been demonstrated. Changing the PL excitation energy provides additional tuning of the photoemission color in the Alq$_3$–PS structures within the range of 515–620 nm.

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Conflict of interest. The authors declare that they have no competing interests.

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Анотація. *У цій роботі досліджено можливість налаштування кольору фотоемісії гібридних структур, отриманих шляхом термічного осадження тонкої плівки трис(8-гідроксихіноліну) алюмінію на поверхню фотолюмінесцентного пористого кремнію. Встановлено, що осаджена плівка є достатньо прозорою у видимому спектральному діапазоні, але частково поглинає УФ-випромінювання. Спектри збудження та випромінювання фотолюмінесценції отриманих гібридних структур досліджували в діапазонах 220–400 та 450–800 нм відповідно. Продемонстровано можливість зміни співвідношення інтенсивностей зеленої та червоної смуг фотоемісії в гібридній структурі шляхом контролю товщини металоорганічної плівки та зміни енергії збудження фотолюмінесценції.*

Ключові слова: *пористий кремній, трис(8-гідроксихінолін)алюміній, гібридна структура, фотолюмінесценція, світлопропускання*