

Photoluminescence of PbS quantum dots in an inorganic glass matrix excited by LEDs: spectra and quantum efficiency

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Received April 29, 2025

Revised September 10, 2025

Accepted October 24, 2025

The luminescent properties of lead sulfide quantum dots grown in a multicomponent silicate glass matrix have been studied to assess the potential of such nanostructured materials for creating broadband near-infrared emitters. In the radiation spectrum of the studied sample, an intense photoluminescence band is observed with a maximum at a wavelength of $\lambda = 1170$ nm and a width at half the height of $\Delta\lambda_{0.5} = 170$ nm. The quantum yield of photoluminescence when excited in the blue region of the spectrum was at least 20%, which can be considered sufficient for competitive near-infrared radiation sources.

Keywords: PbS, quantum dots, glass matrix, photoluminescence, quantum efficiency.

DOI: 10.61011/EOS.2025.10.62562.7931-25

Introduction

Modern optoelectronic devices — light-emitting diodes (LEDs), laser diodes (LDs), solar cells, and others — are based on nanomaterials of various dimensionalities (quantum wells, wires, dots) obtained by different synthesis methods [1]. Among them, glasses and colloidal solutions with quantum dots (QDs) based on binary semiconductors PbS, ZnSe, CdSe, and others, collectively covering the visible and near-IR spectral ranges with their characteristics, are of particular interest. For the latter, PbS QDs in glass are especially noteworthy, as they can exhibit intense photoluminescence spectrally tunable in the wavelength range 1–2 μm depending on QD size, when excited in the visible spectral region by highly efficient LEDs [2]. Obviously, the critical parameter for creating such hybrid emitters, which include pump LEDs and a radiation converter based on PbS QD glass, is the quantum yield of photoluminescence (QY) of QDs in the glass matrix, whose evaluation was this work's aim.

The experimental determination of the QY is complicated by the need to separately measure the total power of the exciting light absorbed by the QD and the total power of the photoluminescence of the QD, which lie in different spectral ranges (visible and IR), with subsequent conversion into the number of quanta taking into account the shape of the spectra. For this reason, it is mostly preferred to use QY standards and perform relative measurements of photoluminescence of the test substance and the standard under the same excitation, and, knowing the corresponding

quantum yield value for the standard, determine the QY value of the test compound [3].

The goal of the work, the results of which are presented in this article, was to measure the absolute QY value in a glass sample with PbS QDs upon photoluminescence excitation by a semiconductor emitter at a wavelength of $\lambda = 460$ nm using an integrating sphere.

Materials and methods

Samples of glasses with PbS QDs were fabricated using the traditional method of growing crystalline phase particles in the bulk of a glass-forming matrix during diffusive phase separation of supersaturated solid solutions [4]. The composition and synthesis procedure of sodium-silicate glasses with lead oxide and sulfur additives, as well as the temperature-time regimes for precipitating lead sulfide crystalline particles in the glass-forming matrix bulk, are described in [5,6]. These same works discuss in detail the structural characterization data of heat-treated samples by small-angle X-ray scattering. It was shown that using two-stage heat treatments allows varying both the volume fraction of the precipitated phase and the average size of grown particles, achieving low size dispersion under certain conditions.

The sample studied in this work was prepared at the initial stage of the phase separation process, when the size of growing particles increases proportionally to the square root of the heat treatment time (shown by the dashed line in Fig. 1, *a*). The particle growth kinetics for the chosen heat treatment regime was adapted from work [5].

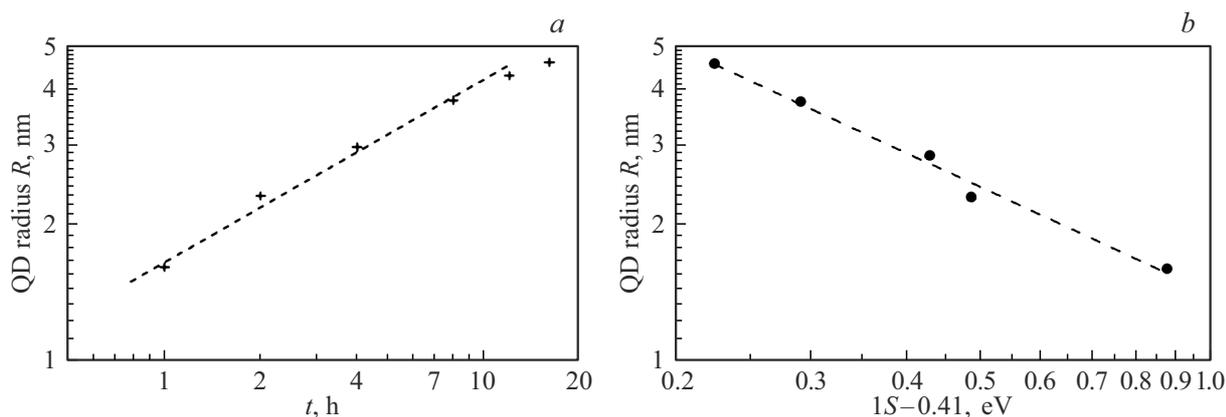


Figure 1. *a* Kinetics of PbS QD growth in sodium-silicate glass. The axis *X* shows the time of secondary heat treatment of samples at $T = 550^{\circ}\text{C}$; *b*) calibration plot for estimating the radius of grown QDs from the position of the absorption band maximum. The QD radius in the studied sample is 1.7 nm.

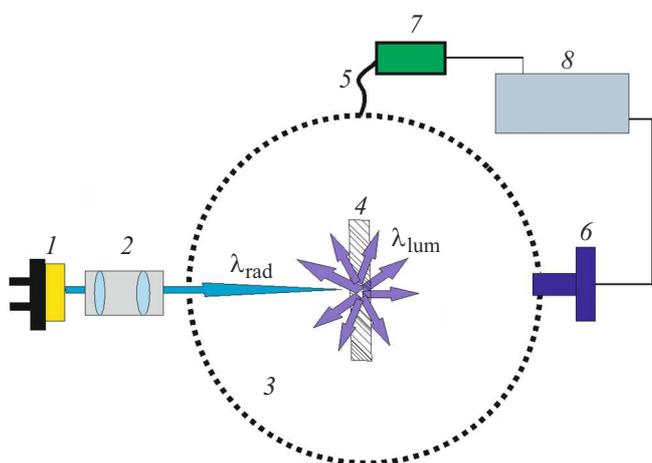


Figure 2. Schematic of the QY measurement experiment: 1 — LD, 2 — collimator, 3 — integrating sphere UPB-150-ART, 4 — test sample, 5 — optical fiber, 6 — power meter XLP12-3S, 7 — UV-VIS photodetector, 8 — spectro radiometer CAS-140.

The average size of grown QDs was estimated from the position of the exciton absorption line maximum $1S$ using the calibration curve (Fig. 1, *b*) $R = f(D)$, where $\Delta = E_{1S} - E_g$ is the shift of the QD absorption edge relative to the fundamental absorption edge of bulk PbS crystals: 0.41 eV ($3\mu\text{m}$) [7]. The calibration plot was obtained from absorption spectra measured for this same series of samples using a Shimadzu UV 3600 spectrophotometer.

Absorption spectra measurements of PbS QDs in glass in the range $400\text{--}1100\text{ nm}$ were performed using the OL770 LED Test and Measurement System (Optronic Lab, USA), which allows obtaining the spectral dependence of the absorption coefficient and the total (absolute) absorbed excitation power from LEDs [8].

Photoluminescence efficiency (down conversion) measurements (Fig. 2) were performed in an UPB-150-ART integrating sphere (Gigahertz-Optik, Germany). The test sam-

ple with dimensions $12 \times 12 \times 2.15\text{ mm}$ was placed at the center of the integrating sphere perpendicular to the beam of collimated LED- or LD-radiation at wavelength $\lambda = 460\text{ nm}$ introduced through the input window corresponding to 0° according to the sphere calibration. The divergence ensured the beam reached the output (opposite, 180-degree) window of the sphere, onto which a thermoelectric power meter XLP12-3S (Gentec Electro-Optics Inc.) was installed. To the output window corresponding to 90° of the sphere, a CAS-140 spectro radiometer (Instrument systems, Germany) with optically independent UV-VIS ($250\text{--}820\text{ nm}$) and IR ($780\text{--}1650\text{ nm}$) modules was connected via an optical fiber cable. The spectro radiometer provided registration of the emission spectrum and power in relative units. An important advantage of such a spectro radiometer for solving the discussed experimental task was the optical independence of the UV-VIS and IR modules when registering parameters of the initial (460 nm) radiation and photoluminescence initiated in the sample. This allowed evaluating the photoluminescence level without influence from background radiation arising in the integrating sphere due to scattering and reflection of the exciting beam by the sample.

Results and discussion

The absorption and photoluminescence spectra of PbS QDs in glass are shown in Fig. 3. The absorption spectrum has a maximum of the first absorption band at $\lambda = 1040\text{ nm}$, and then the absorption coefficient monotonically increases when shifting toward shorter wavelengths, reaching values of $\alpha \sim 60\text{ cm}^{-1}$ at $400\text{--}500\text{ nm}$. The photoluminescence spectrum has a maximum at wavelength $\lambda_{\text{max}} = 1170\text{ nm}$ and a full width at half maximum of $\Delta\lambda_{0.5} \approx 170\text{ nm}$.

The QY calculation η was performed based on measurements of absorbed power ϕ_{abs} and photoluminescence power ϕ_{lum} and calculation of the number of quanta based on their average energy from the spectrum $h\bar{\nu}$.

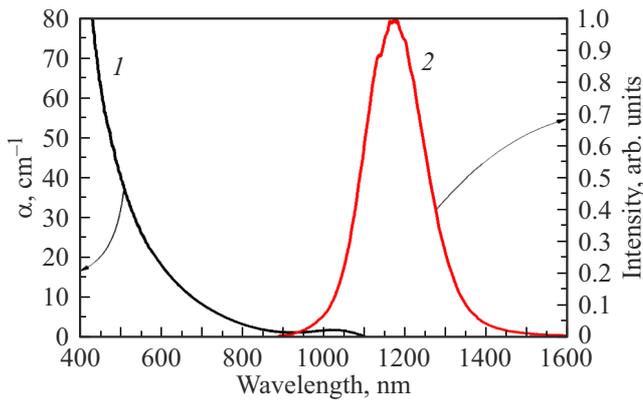


Figure 3. Absorption (1) and photoluminescence (2) spectra of PbS QDs.

The number of quanta in an elementary spectral interval $d\lambda$ of spectrum $\phi_{e,\lambda}$ is equal to

$$n = \frac{\phi_{e,\lambda}}{h\nu} = \frac{\phi_{e,\lambda}\lambda}{hc}. \quad (1)$$

The total number of quanta in the spectrum

$$N = \int \frac{\lambda}{hc} \phi_{e,\lambda} d\lambda. \quad (2)$$

And then the average quantum energy

$$h\nu = \phi_e \int (\lambda \phi_{e,\lambda} / hc) d\lambda, \quad (3)$$

where ϕ_e is the total power over the spectrum.

Thus, the number of quanta in the absorbed radiation and photoluminescence fluxes

$$N_{\text{abs}} = \frac{\phi_{\text{abs}}}{h\nu_{\text{abs}}}, \quad N_{\text{lum}} = \frac{\phi_{\text{lum}}}{h\nu_{\text{lum}}}, \quad (4)$$

and the photoluminescence quantum yield

$$\eta = \frac{N_{\text{lum}}}{N_{\text{abs}}} = \frac{\phi_{\text{lum}}/h\nu_{\text{lum}}}{\phi_{\text{abs}}/h\nu_{\text{abs}}}. \quad (5)$$

ϕ_{abs} at sufficiently high medium absorption ($\alpha \sim 60 \text{ cm}^{-1}$) equals the LD excitation power P_{out} .

In the experiment, the number of absorbed excitation light quanta from the LD at $\lambda = 460 \text{ nm}$ was $N_{\text{abs}} = 2.73 \cdot 10^{17}$, and the number of photoluminescence quanta was $N_{\text{lum}} = 5.37 \cdot 10^{16}$. The obtained QY value $\eta \approx 20\%$ should be considered acceptable for practical applications with prospects for increase upon improvement of PbS QD technology in glasses. Note that the obtained value may also be slightly underestimated considering that not all photoluminescence radiation leaves the sample.

Thus, by integrating a glass sample containing PbS QDs with a semiconductor LED or LD emitter, an efficient near-IR range radiation source can be obtained. In this case, the emission wavelength maximum can be controlled by varying the QD size during their synthesis in the glass matrix.

Acknowledgments

The studies were performed at the common use center „Hardware Components of Radio Photonics and Nanoelectronics: Technology, Diagnostics, Metrology“.

Conflict of interest

The authors declare that they have no conflict of interest.

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Translated by J.Savelyeva