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# The heat treatment effect on the spectral and luminescent properties of sodium-germanate glass with CdS

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## ABSTRACT

The nucleation of CdS quantum dots in a sodium germanate glass matrix by means of isothermal treatment is demonstrated. It is shown that a sufficiently high concentration of cadmium ions in the initial glass results in nucleation of fairly big quantum dots. The luminescence of CdS is mainly of a trap nature and is localized in the region of 600–900 nm. Excess cadmium ions and nucleation of quantum dots in oxide glass lead to the defect nature of the luminescence bands: mainly associated with interstitial cadmium ions (ICd), the donor-acceptor pairs  $[V_{Cd}-V_S]$ , and deep trap levels created by volume cadmium vacancies. The luminescence quantum yield of CdS quantum dots is less than 1 %, indicating that nonradiative recombination of charge carriers is the dominant process.

## KEYWORDS

cadmium sulfate • germanate glass • heat-induced crystallization • trap luminescence

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## Introduction

CdS quantum dots (QDs) have attracted much attention as a special class of nanoparticles due to their luminescent and semiconducting properties [1–3]. Due to the intense luminescence, continuous excitation spectrum, controllable emission band location, and ease of functionalization for tissue labeling, CdS QDs have been becoming promising for medical imaging and disease treatment [4–6]. Since CdS has a wide band gap, it is used as a window material for heterojunction solar cells, avoiding the recombination of photogenerated charge carriers and improving the efficiency of solar cells [7]. Due to its high photostability, CdS can be a basis for light-emitting diodes, photodetectors, and sensors [8,9].

The photoluminescence of QDs is very intense and occurs with a fairly narrow spectral profile: the full width at half maximum (FWHM) is usually less than 40 nm [10]. By choosing the chemical composition (i.e. the band gap of the bulk material) and the size of the nanocrystals, it is possible to tune the radiation energy so that it falls in the spectral region from UV to IR. Optical and electronic processes in a semiconductor nanocrystal include radiative and nonradiative recombination of excitons; radiative and

nonradiative recombination of surface defect states, which in turn can be an oxidizing surface trap (electron trap) and a reducing surface trap (hole trap) [11].

A surface passivation procedure is used to reduce the influence of defects on the optical and spectral properties of QDs and to reduce the probability of nonradiative relaxation of photoexcitation. Quite a few methods of surface passivation have been considered to date. In this paper, we will focus on the nucleation of quantum dots in an inorganic glass matrix [12,13], which was initially intended as protection of the QD surface from interaction with an oxygen-containing atmosphere and stabilization of the quantum dot structure [14–16].

As off today, only three works have been devoted to the nucleation of cadmium chalcogenide quantum dots in germanate glass [17–19], but none of them demonstrate the luminescent and optical properties of the resulting nanocrystals. Alkali metal oxides are usually added to pure  $\text{GeO}_2$  [20–22] to improve the crystallization ability of the germanate glassy matrix. Since glass synthesis is usually carried out in a reducing atmosphere to decrease sulfur volatility, the use of lithium ions in large quantities is contraindicated. Therefore, sodium oxide was used in the germanate matrix to nucleate cadmium chalcogenide nanocrystals [17].

Here, the influence of the isothermal treatment mode on the spectral and luminescent properties of CdS nanocrystals nucleated in a sodium germanate glass matrix is demonstrated.

## Methods

The chemical composition of initial sodium-germanate glass matrix was: 20  $\text{Na}_2\text{O}$  – 80  $\text{GeO}_2$  mol. %, 7.4 mol. % CdS were introduced as activating additives over 100 % of the matrix. Chemicals  $\text{Na}_2\text{CO}_3$ ,  $\text{GeO}_2$ , CdS of reagent grade were used for synthesis of 100 g glass bulk. Glasses were synthesized in a Gero laboratory high-temperature furnace at 1150 °C for 30 min in closed quartz crucible by standard melt-quenching technique in argon atmosphere. In two articles devoted to the nucleation of CdS and CdSe in sodium germanate glass, the authors took either 8  $\text{Na}_2\text{O}$  – 92  $\text{GeO}_2$  (mol. %) composition with subsequent heat treatment (HT) at a temperature of 500–550 °C [18], or 40  $\text{Na}_2\text{O}$  – 60  $\text{GeO}_2$  (mol. %) composition with heat treatment at 580–620 °C [17]. Since in this work an intermediate composition in terms of  $\text{Na}_2\text{O}$  content was chosen, subsequent single-stage heat treatment was conducted in the Nabertherm muffle furnace at 560 °C.

Absorption spectra were measured on a double-beam Lambda 650 PerkinElmer spectrophotometer in the wavelength range 200–800 nm with 1 nm resolution. For these measurements polished plane samples of 0.8–1.0 mm thick were prepared.

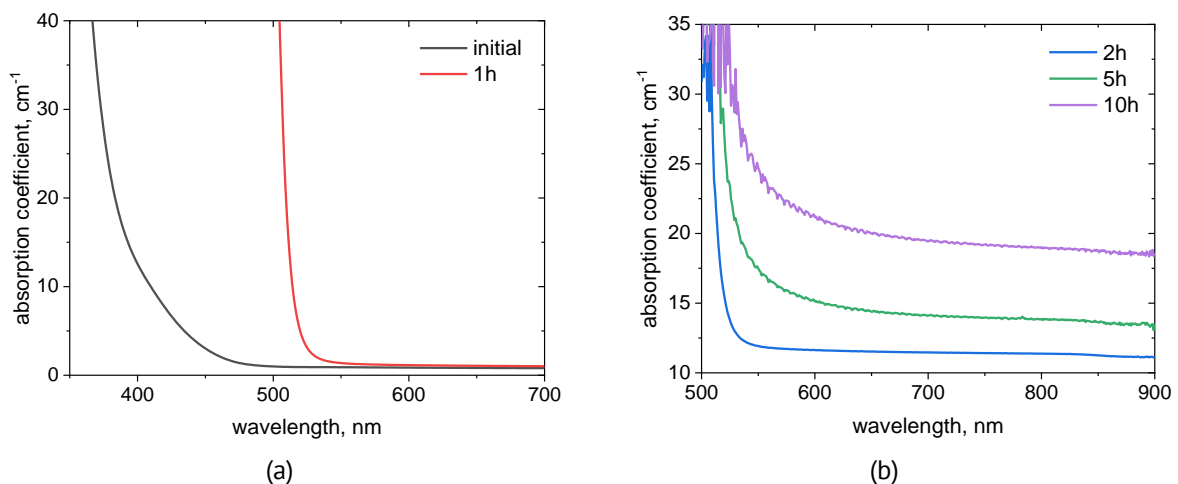
Photoexcitation and photoluminescence spectra were obtained by spectrofluorometer LS-55 (Perkin Elmer) in the 200–900 nm region with 1 nm step. The luminescence decay kinetics were obtained using a pulse mode of a built-in Xenon lamp and by varying the time delay from the exciting pulse with a step of 0.01  $\mu\text{s}$ . Based on this, the time dependence of the luminescence intensity was plotted and mathematically processed by the Origin Pro software.

The actual chemical composition of the glass-ceramics was determined by X-ray fluorescence spectroscopy (XRF) using an X-ray fluorescence spectrometer ARL

PERFORM'X by Thermo Scientific with Rh tube, 4.2 kW/60kV generator, FPC and Sc detectors (chemical elements: from oxygen to uranium). The study was done at ambient temperature in the vacuum atmosphere. The chemical composition was averaged over a sample area of 20 mm<sup>2</sup>. The resulting composition of the glass under study was: 7.4 Na<sub>2</sub>O – 84.8 GeO<sub>2</sub> – 7.5 CdO – 0.3 S wt. %. Despite the use of a closed crucible and an inert atmosphere, the sulfur content decreased by 4 times due to high volatilization. According to the phase equilibrium diagram of the Na<sub>2</sub>O-GeO<sub>2</sub> system [23], in the region of high GeO<sub>2</sub> content, the compounds Na<sub>2</sub>O·4GeO<sub>2</sub> or 2Na<sub>2</sub>O·9GeO<sub>2</sub> should crystallize. The eutectic point between first compound and GeO<sub>2</sub> accounted for 94.5 wt. % GeO<sub>2</sub> having a melt temperature of 950 °C. Since in our case the actual Na<sub>2</sub>O/GeO<sub>2</sub> ratio was close to this numbers, we should expect the corresponding phase separation.

## Results and Discussion

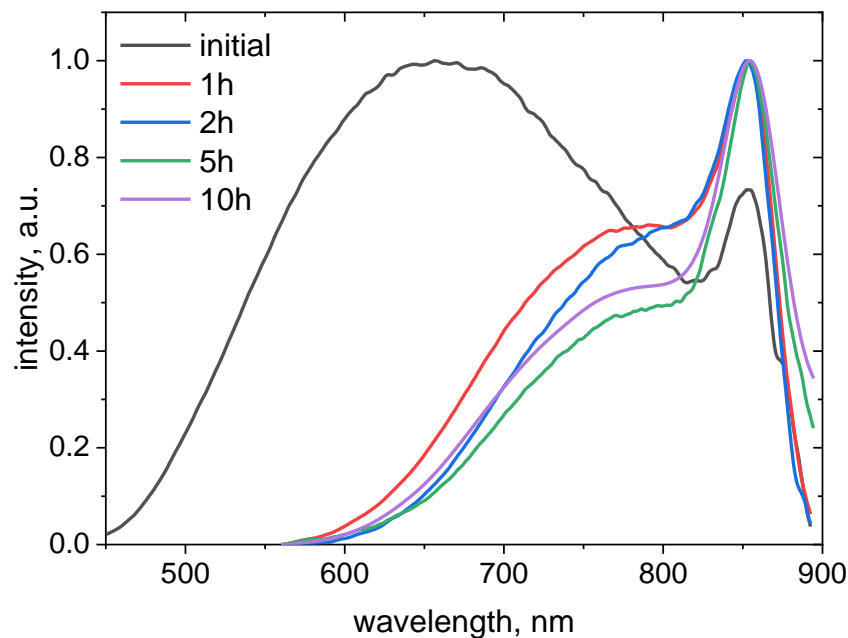
Figure 1 shows the absorption spectra of the initial glass and glass-ceramics after the heat treatment at a temperature of 560 °C for different durations. The heat treatment temperature was chosen according to the literature and below the exothermic peak on the differential scanning calorimetry curve for the glass matrix under study [24]. Nevertheless, even a duration of 1 hour led to intensive release of CdS nanocrystals in the glass matrix (Fig. 1(a)). This was confirmed by the fact that the absorption edge of the initial glass located in the region of 400 nm (3.1 eV), and the absorption boundary of the heat-treated glass was in the region of 512 nm (2.42 eV). The band gap of bulk CdS in the form of zinc blende is 2.42 eV [25]. Thus, after the heat treatment, rather large crystals were immediately released in the glasses under study. With an increase in the heat treatment duration, the amount of the crystalline phase increased, which was confirmed by an increase in the absorption intensity of nanocrystals. As well as the scattering level was increased which was associated with phase separation and partial crystallization of the glass matrix. In this case, the absorption edge location remained practically unchanged, which indicated that the mean crystal size remained the same.



**Fig. 1.** Absorption spectra of the glass under study: initial and after heat treatment at a temperature of 560 °C with different durations

It is worth noting that the location of the band gap edge on the absorption spectrum is directly related to the size of the quantum dot [26–28]. If the position of the absorption edge is close to the band gap of the macrocrystal ( $E_{\text{ex}} = 2.4 \div 2.5$  eV), then quantum dots have grown so that they are either related to the weak size quantization regime [29,30] or no longer obey the rules of strong size quantization [31,32] (in case of exceeding the Bohr exciton radius  $a_{\text{ex}} = 2.5$  Å [33,34] by two times).

For the exciton photoluminescence band of CdS QDs, the Stokes shift was in the range of 0.01–0.15 eV with a band maximum at 350–480 nm for QDs with an average size of 1.5–5 nm [25,35,36]. For the luminescence band associated with radiative recombination involving defects, the Stokes shift was significantly larger and varied from 0.2 to 1.2 eV for different defects [25,35,36]. The presence of defects leads to the appearance of localized levels within the band gap. Even though at low concentrations of defects these levels are practically not distinguished in the absorption spectra, their contribution to luminescence can be significant, as in our case (Fig. 2).



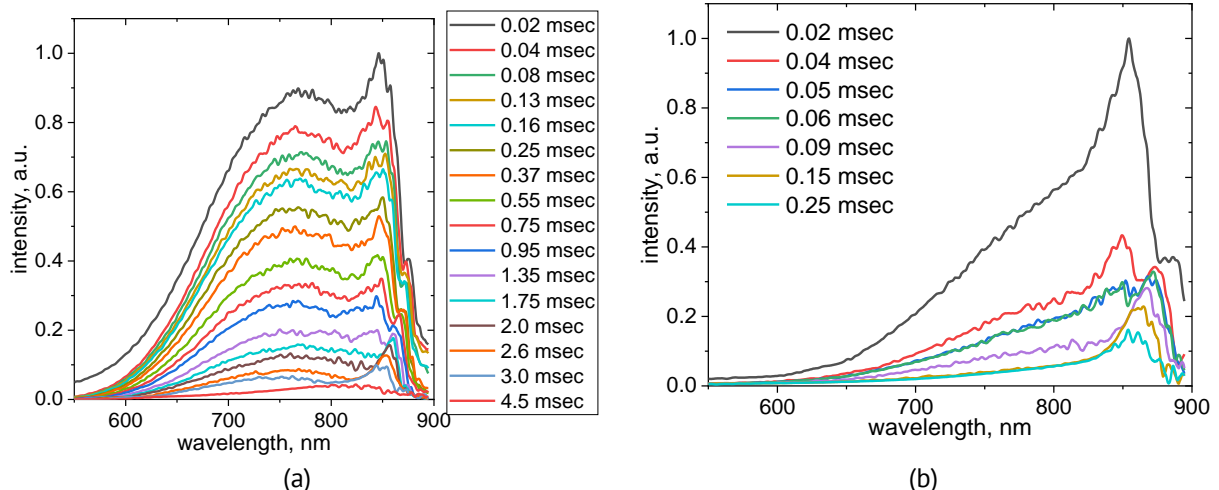
**Fig. 2.** Luminescence spectra of the studied glass: initial and after heat treatment at a temperature of 560 °C with different durations ( $\lambda_{\text{ex}} = 390$  nm)

The luminescence of CdS QDs in the long-wavelength region is determined by the local states associated with intrinsic defects of the crystal structure. Such defects are surface states, interstitial cadmium and sulfur ions ( $I_{\text{Cd}}$ ,  $I_{\text{S}}$ ), sulfur vacancies ( $V_{\text{S}}$ ), cadmium vacancies (single  $V_{\text{Cd}}$ , paired with a sulfur vacancy [ $V_{\text{Cd}}-V_{\text{S}}$ ] or oxygen in the sulfur position [ $V_{\text{Cd}}-O_{\text{S}}$ ]) [16,25,37–39]. According to these papers, in single crystals cadmium vacancies  $V_{\text{Cd}}$  led to the luminescence at 1.2 eV, the complex of a cadmium vacancy with oxygen [ $V_{\text{Cd}}-O_{\text{S}}$ ] – 1.51 eV, the donor-acceptor pair [ $V_{\text{Cd}}-V_{\text{S}}$ ] – 1.72 eV, the interstitial cadmium atom  $I_{\text{Cd}}$  – 2.05 eV, the interstitial sulfur atom  $I_{\text{S}}$  – 2.38 eV and sulfur vacancies  $V_{\text{S}}$  – in the region of 1.77–1.85 eV.

Due to the presence of several factors, namely: the quantum-size effect, size dispersion, and the allocation of quantum dots in the oxide glass matrix, in our case the location of the corresponding bands will differ slightly from those indicated above. A wide structureless band, which dominated the luminescence spectrum (Fig. 2) with a maximum at 650 nm was a superposition of at least three bands with maxima at 610, 700, and 765 nm. These bands can be attributed to transitions to levels associated with interstitial cadmium ions ( $I_{Cd}$ ), and the donor-acceptor pair  $[V_{Cd}-V_S]$ . A separate band with a maximum at 855 nm can be associated with a radiative transition involving the states of the cadmium vacancy with oxygen  $[V_{Cd}-O_S]$ . An increase in the intensity of this band after isothermal treatment confirmed its oxygen nature, since the heat treatment was carried out in an oxygen-containing atmosphere. According to the analysis of the chemical composition, an excess of cadmium ions over sulfur was found in the glass under study, thus the probability of the presence of many cadmium vacancies and interstitial cadmium ions was quite high; therefore, most of the radiative transitions were attributed to these types of structural defects.

The luminescence quantum yield in the studied glasses was less than 1 %. Low QY is usually taken as a sign that nonradiative recombination of charge carriers is the dominant process. Since in our case the luminescence spectra demonstrate a large number of bands associated with structural defects, the probability of nonradiative recombination on them can be quite high.

The multi-exponential luminescence decay observed for cadmium chalcogenide nanocrystals also indicates the distribution of both the size and shape of the nanoparticles, the presence of radiative states with different lifetimes, as well as the release of traps and the redistribution of charge carriers at different energies [40–42].



**Fig. 3.** Time-resolved luminescence spectra of the initial glass (a) and glass after heat treatment for 2 hours (b) with different delays after the exciting pulse ( $\lambda_{ex} = 390$  nm)

The time-resolved luminescence spectra for the initial and heat-treated glass are shown in Fig. 3. The luminescence decay kinetics of the heat-treated sample was described by a single-exponential function with a lifetime of 17  $\mu$ sec, which corresponded to the decay of a Xenon flash lamp used in the registration method. This

suggests that the decay kinetics of CdS in this case was probably quite fast: on the order of hundreds of nanoseconds, which is quite consistent with the literature [43,44] and which is not resolvable in this method. However, the luminescence decay kinetics of the initial glass with CdS was significantly more protracted and the single-exponential lifetime was 850  $\mu\text{sec}$  at 760 nm and 662  $\mu\text{sec}$  at 845 nm. The nature of such a long lifetime requires additional research. In both graphs (Fig. 3), after a certain delay, a luminescence band with a maximum at 875 nm appeared in the spectra, which can be attributed to transitions to deep trap levels created by cadmium vacancies in the volume of nanocrystals.

## Conclusions

The article demonstrated the nucleation of CdS quantum dots in a sodium germanate glass matrix by means of isothermal treatment, as well as the dependence of the luminescent properties of quantum dots on the treatment mode. It was shown that due to a sufficiently high concentration of cadmium ions in the initial glass components, the resulting quantum dots were quite large. The luminescence of CdS was mainly of a trap nature and was localized in the region of 600–900 nm. Excess cadmium ions and nucleation of quantum dots in oxide glass led to the fact that the nature of the luminescence bands was mainly associated with such crystal structure defects as interstitial cadmium ions ( $I_{\text{Cd}}$ ), the donor-acceptor pairs [ $V_{\text{Cd}}-V_{\text{S}}$ ], and deep trap levels created by volume cadmium vacancies. The luminescence quantum yield of CdS quantum dots was less than 1 %, indicating that nonradiative recombination of charge carriers was the dominant process.

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