

Semiconductor doped glasses

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This paper reviews some of the interesting applications of semiconductor nanocrystals embedded in glasses. Because of their unique electronic and optical properties, semiconductor quantum dots (QDs) found wide applications in photonics. They are inexpensive and robust material and have attracted a great attention in the application of 1.3–1.5 μm optical communication. Embedding semiconductor QDs in spherical microcavities is also an attractive system both for fundamental physics research in the field of cavity quantum electrodynamics and for its potential application in lasing materials. Energy transfer processes and coupling-strength between nanocrystals can be studied and demonstrated using semiconductor nanocrystals doped glasses. It has been established that an efficient energy transfer occurs between QDs having a significant band gap energy difference, i.e., between dots of markedly different sizes. We demonstrate that a promising way to enhance energy transfer between different subsets of QDs is to red-shift the emission sufficiently far from the host absorption so that self-absorption by the host becomes negligible.

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1. Introduction

Semiconductor nanocrystals (or quantum dots, QDs) represent a general class of materials that are intermediate between bulk materials and molecular species. Because of the quantum confinement effect, semiconductor nanocrystals exhibit size dependence, molecular-like discrete electronic and optical transitions.^{1,2)} There has been considerable research in this field, especially in the optical properties of semiconductor QDs. A number of matrices have been used for the preparation of semiconductor nanoparticles including glasses. These matrices can be viewed as nanochambers that limit the size to which crystallites can grow.³⁾ The properties of the nanocrystallites are determined, not only by the confinements of the host material but also by the properties of the system, which include the internal/external surface properties of the nanocrystallites. New nonlinear composite materials can be prepared by doping glasses with semiconductor nanocrystallites.⁴⁾ By adding their unique electronic and optical properties, semiconductor QDs found wide applications in the optoelectronics. For instance, semiconductor QDs from the IV–VI family, such as PbS, PbSe, provide access to the limit of strong quantum confinement effect, compared to II–VI or III–V semiconductors, and thus offer excellent opportunities for both experimental and theoretical investigations.^{5,6)} They are interesting for infra-red application due to their narrow band gap and large exciton Bohr radii.

The large surface area of a nanocrystal and the surrounding medium, such as the capping agent, can have a profound effect on the properties of the particles. Defects within the nanocrystal act as electron/hole traps that can lead to nonlinear optical effects.⁷⁾ Thus, many strategies have been used to coat the surface of the QDs with glass.⁸⁾ These types of semiconductor doped glass have an improved stability against flocculation, increased quantum yield, and surface ligands. The structural characterization is also of interest for a better understanding of

the physical processes underlying the growth of crystallites inside the glass matrix. The average size of nanocrystals can be tailored by controlling the temperature or time of treatment. The major problem is the size dispersion of the crystallites, which is intrinsic to the diffusion process.

2. Structural and intrinsic properties

The first results on the synthesis of molten glass containing PbS and PbSe crystallites were reported by Borrelli in 1994.⁹⁾ Later, the synthesis of phosphate glasses containing PbSe nanocrystals,¹⁰⁾ phosphate, silicate, and boron-silicate glasses with PbS nanocrystals,^{11,12,13)} and the synthesis of silicate glasses containing nanocrystals of PbTe were demonstrated as well. **Figure 1** demonstrates the room-temperature absorption spectra of PbS QD doped glasses with different QD radii. One of the most important advantages of semiconductor-doped glasses is the low cost to fabricate compared to epitaxially grown

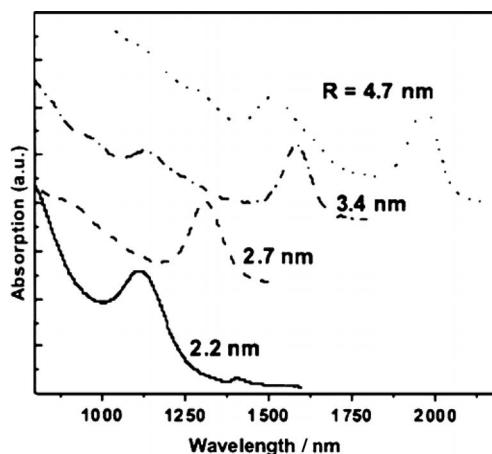


Fig. 1. Room-temperature absorption spectra of PbS QD-doped glasses with mean QD radii R .¹³⁾

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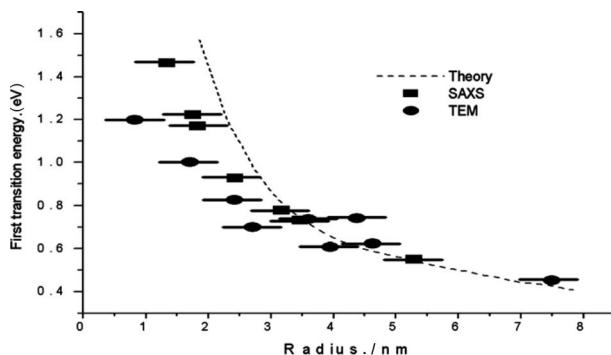


Fig. 2. Squares and circles show the first exciton transition energies plotted against radii obtained from SAXS and TEM data.¹⁶⁾

techniques such as molecular beam epitaxy. Suitable thermal treatment of a glass containing the chemical components of a semiconductor can precipitate semiconductor QDs with narrow-size distributions with nice properties, such as few substitutional defects, and few dangling bonds. Jain and Lind have prepared nanocrystals of CdS or CdSSe semiconductors embedded in glass matrix.¹⁴⁾ These semiconductor-doped glasses samples have demonstrated large optical nonlinearity and a fast response time. Other glass systems have also demonstrated quantum confinement with microcrystallite CdTe semiconductors borosilicate glasses. The microcrystallite sizes are controlled by the heat-treatment time and temperature.¹⁵⁾ The size of the QDs can be evaluated using small angle X-ray scattering (SAXS).¹⁶⁾ However, the only direct method to observe the shape of the QDs and to evaluate their size distribution is transmission electron microscopy (TEM).¹⁷⁾ These techniques are used to allow collecting the main information about the QDs embedded in glass matrix: their chemical composition, crystalline structure, size, and size distribution. For the electronic characterization many techniques have been used exhaustively, such as optical absorption techniques,¹⁸⁾ luminescence spectroscopy, Raman spectroscopy,¹⁹⁾ and measurements of nonlinear optical response using time-resolved luminescence.²⁰⁾

The most striking property of semiconductor nanocrystals is the massive changes in the optical properties as a function of the nanocrystal size.²¹⁾ As the size is reduced, the electronic excitations shift to higher energy, according to **Fig. 2**, and there is concentration of oscillator strength into just a few transitions. These basic physical phenomena of quantum confinement arise by changes in the density of electronic states and can be understood by considering the relationship between position and momentum in free and confined particles. For a free particle or a particle in a periodic potential, the energy and the crystal momentum p_k may both be precisely defined, while the position is not. As a particle is localized, the energy may still be well-defined; however, the uncertainty in position decreases, so that momentum is no longer well-defined. The energy eigenfunctions of the particle may then be viewed as superpositions of bulk k -states. In the extended case, there is a relationship between energy and momentum, and to a first approximation, the change in energy as a function of the size can be estimated simply by realizing that the energy of the confined particle arises by superposition of bulk k -states of differing energy.

There are two major effects that are responsible for the size variations in nanocrystal properties. First, in nanocrystals the number of surface atoms is a large fraction of the total. Second,

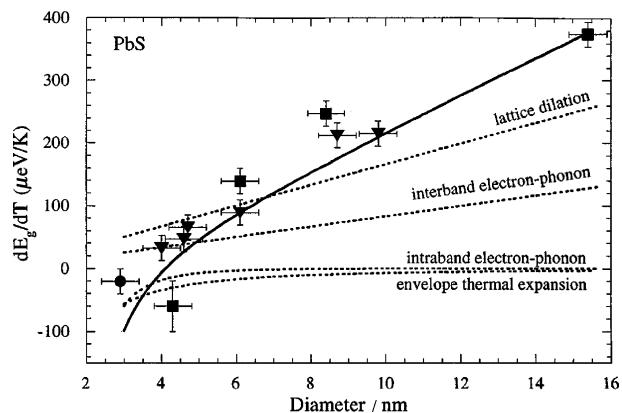


Fig. 3. Temperature coefficients of electron-hole pair energies dE/dT in PbS quantum dots as a function of the size of the quantum dot. Symbols are experimental values. Squares: phosphate glass host; triangles: oxide glass host; circle: polymer host. Calculated contributions to dE/dT are shown as dotted lines, and the solid line is the sum of all contributions.²³⁾

the intrinsic properties of the interior of nanocrystals are transformed by quantum size effects. In any material, surface atoms make a distinct contribution to the free energy, and the large changes in thermodynamic properties of nanocrystals (melting temperature depression, solid-solid phase transition elevation) can ultimately be traced to this. The QD radii R quoted in Fig. 2 can be calculated using a hyperbolic band model:

$$(\hbar\omega_{ls})^2 = \left(\frac{hc}{\lambda_{ls}}\right)^2 = E_g^2 + \left(\frac{2\hbar^2 E_g}{m^*}\right) \left(\frac{\pi}{R}\right)^2,$$

by using the room temperature band gap energy of $E_g = 0.41$ eV and the effective mass of $m^* = 0.12 m_0$ for PbS.²²⁾

At the surface of a pure semiconductor nanocrystal, substantial reconstructions in the atomic positions occur, and invariably energy levels lie within the energetically forbidden gap of the bulk solid. These surface states act as traps for electrons or holes and degrade the electrical and optical properties of the material. The chemical process by which these surface atoms are bonded to another material of a much larger band gap is passivation, in such a way as to eliminate all the energy levels inside the gap. The ideal termination naturally removes the structural reconstructions and simply produces an atomically abrupt jump in the chemical potential for electrons or holes at the interface.

Another important feature related to the nanocrystal QDs is the temperature dependence of the gap energy, i.e., dE/dT . **Figure 3** shows that the temperature coefficients of electron-hole pair energies in PbS and PbSe quantum dots depend strongly on the size of the nanocrystal QD. By decreasing the QD size the temperature coefficient of the lowest electron-hole pair energy dE/dT decreases by more than an order of magnitude from the bulk value. The contributions from interband electron-phonon, intraband electron-phonon, envelope thermal expansion, and lattice dilatation are taken into account. Note that dE/dT becomes slightly negative for smaller QDs according to A. Olkhovets et al.²³⁾

3. Applications

3.1 Nonlinear phenomena

Semiconductor-doped glasses embedding nanocrystals of CdS or CdSSe demonstrates large optical nonlinearity and a fast response time.²⁴⁾ However, the response time in these glasses

decreases by light irradiation, a process known by photo-darkening effect. Photo-darkening is a photo-chemical process and involves deposition of ions on the existing QDs. This apparent increase in the size of the QDs leads to reduction in nonlinear optical properties, which limits the applications of nanocrystals towards opto-electronic devices.²⁵⁾ Results also indicate that defects are created by laser irradiation. The photoinduced defects are considered to be due to trapped electrons in the glass matrix.^{26),27)} So, they act as nonradiative recombination centers^{28),29)} and provide additional channels for the recombination of excited carriers, increasing the decay rate of the luminescence.

After 1997, substantial progress occurred and quite impressive results were obtained, mainly due to the fabrication of glasses colored with narrow band-gap IV-VI semiconductors, the case of PbS and PbSe.^{9),30)} The band-gap wavelengths of PbS and PbSe are approximately 3.0 and 4.5 μm , respectively, at room temperature. These might allow exciton absorption band in the wavelength range 1–3 μm . Glasses containing PbS or PbSe nanoparticles of different sizes were applied as saturable absorbers for near-IR lasers.¹⁶⁾ This means that these materials can be used for both mode-locking and Q-switching of near-infrared solid-state lasers emitting at the wavelengths from 1.0 to 2.1 μm . Passive shutters based on saturable optical absorption allow obtaining light pulses of high power and short/ultrashort duration.

The nonlinear optical properties of PbS QD-doped glasses have been studied recently for mode-locking³¹⁾ and gain³²⁾ applications; however, all of these studies have been performed in bulk glasses. Waveguides produced by using semiconductor doped glasses have been produced for integrated photonic devices.³³⁾ CdTe-doped glass has also demonstrated to be able to passively mode lock a Ti:sapphire laser.³⁴⁾

Various nonlinear optical techniques, i.e., degenerate four wave mixing (DFWM),³⁵⁾ non-degenerate four wave mixing,³⁶⁾ spectrally resolved two beam coupling technique³⁷⁾ and single beam Z-scan³⁸⁾ have been employed to measure the nonlinear optical response of semiconductor doped glasses containing nanocrystals of semiconductors. Miyoshi et al. investigated the femtosecond dynamics in semiconductor-doped glasses using DFWM experiments.²⁴⁾ They demonstrated two main contributions for the signal: the fast contribution was attributable to the glass matrix, and the slow contribution was attributable to the photogenerated carriers in semiconductor nanocrystals. One of the applications upon the nonlinear optical properties presented in PbS, PbSe, and CuSe nanoparticles embedded in glass regards to the design of saturable absorbers.¹⁶⁾ The nonlinear properties excited under nonresonant below gap condition was investigated by Tsuda and Cruz in CdS_xSe_{1-x} semiconductor-doped glass. They mainly observed the optical Stark shift probed with femtosecond optical pulses.²⁰⁾

3.2 Lasing in semiconductor doped microspheres

Embedding semiconductor QDs in spherical microcavities is an attractive system both for fundamental physics research in the field of cavity quantum electrodynamics and for its potential application in optoelectronics. Artemyev and Woggon prepared spherical microcavities using a chemical way.³⁹⁾ They embedded CdSe semiconductor QDs in the polymethylmethacrylate microsphere and realized the coupling of electronic and photonic states. Instead, an approach to prepare glass microspheres embedded with CdSSe semiconductor QDs, which have a better optical stability than the polymer microspheres, can be used because the latter are often degraded under strong illumination

due to the photosensitive surface reactions. The glass has a refractive index of 1.5 or larger, being suitable to employ as the microcavity media surrounded by the air. When a single glass microspherical cavity is excited by a laser beam at room temperature, very strong and sharp resonance peaks were shown on the PL spectra, which can be well explained by the coupling of QD luminescence with the whispering gallery modes (WGM) of the spherical microcavity.⁴⁰⁾ **Figure 4** shows this strong coupling in the PL spectrum of a single microsphere (4.6 μm in diameter) at room temperature exciting at 488.0 nm.

In order to prepare the optical microspheres containing semiconductor QDs, Jia et al. have used commercially available glass ingots that are a source material for making optical filter glasses, for example RG 645 or other types in the same series.⁴¹⁾ The glass ingots contain CdO, CdS, and elemental sulfur or selenium in their composition. They are pulverized into small glass pieces, with the size of a few micrometers and sifted by sieves. Employing a specially designed gas burner to heat and blow off micrometer-sized glass pieces, the glass pieces melt at a temperature higher than the softening point and form into microspheres due to the surface tension. The obtained microspheres can still be annealed at certain temperature for several hours in order to form precipitates and grow CdSe_xS_{1-x} nanocrystals QDs in the microspheres. The size of the CdSe_xS_{1-x} semiconductor nanocrystals depends on the annealing temperature and time.^{42),43)} In the semiconductor doped glass microspheres lower threshold of stimu-

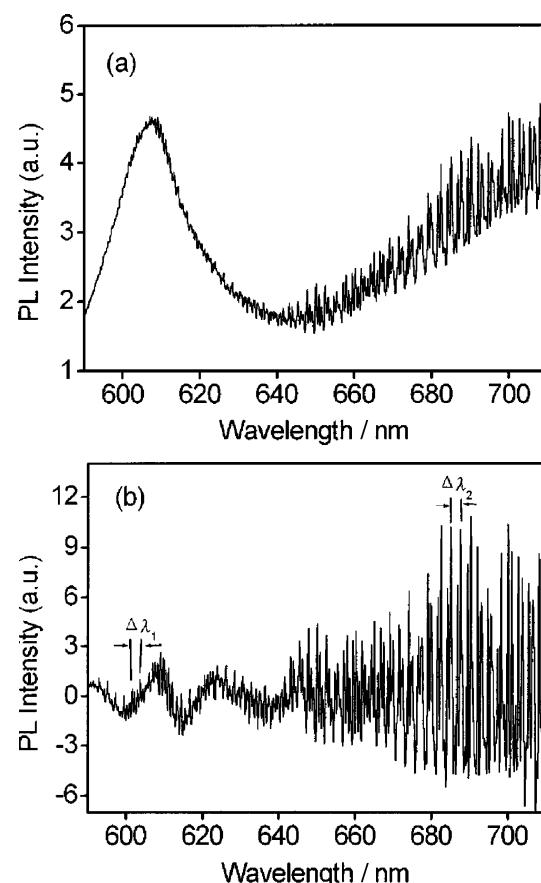


Fig. 4. (a) PL spectrum of a single glass microsphere with CdSe_xS_{1-x} QDs, (b) Normalized PL spectrum after subtracting the luminescence background, showing clearer WGM. Clear separations between the adjacent peak wavelengths of the resonant WGMs are shown by arrows.⁴¹⁾

lated emission or lasing modes of QDs may be realized. Resonant WGMs are observed in the glass microspheres with QDs for the luminescence bands of both excitonic transitions and from surface defect states of the embedded QDs. High Q -values of quality factor are realized for these WGMs as well.

4. Energy transfer mechanisms in PbS and PbSe quantum dots

Communication, coupling, and coherence between quantum dots have appeared as central themes in numerous scientific efforts of present physical and technological interest.^{44),45)} For instance, in strongly coupled QD assemblies, electronic excitations can, in principle, delocalise across multiple dots leading to new states described by coherent superposition of individual dot wave functions.⁴⁶⁾ An experimental study to evaluate the strength of coupling between in PbS and PbSe nanocrystal QDs has been carried out by analyzing the spatially-resolved PL on the sample surface.⁴⁷⁾ The investigation of the carrier transfer dependence against radius clarifies the process of energy transfer between the nanocrystal QDs. The PbS and PbSe nanocrystal QDs were synthesized in oxide glasses. The term “photon diffusion length” is used to determine the strength of interaction among the dots.⁴⁸⁾ This interesting way became useful to characterize nanocrystal samples that contain different assemblies of sizes.

The semiconductor QD glass samples, PbS and PbSe, were fabricated using sulphur (or selenium) doped oxide glass matrix ($\text{SiO}_2\text{--Na}_2\text{CO}_3\text{--Al}_2\text{O}_3\text{--PbO}_2\text{--B}_2\text{O}_3$) prepared from high purity powders using SiO_2 as glass former and Na_2CO_3 to reduce the melting point.¹⁸⁾ The mixture was then melted in an alumina crucible at 1200°C for 30 min. Then, it was cooled down to room temperature. Thermal treatment of the glass matrix was performed at 500°C to enhance the diffusion of Pb^{2+} and S^{2-} (Se^{2-}) ions. As a result of the thermal treatment PbS (PbSe) QDs were formed in the glass matrix.

In Fig. 5, the photoluminescence (PL) and the optical absorption (OA) spectra of PbS and PbSe quantum dots at room temperature can be observed.¹⁸⁾ Using these data nanocrystal sizes were estimated by calculating the exciton energy as a function

of radius for the fundamental state (1sh–1se).⁴⁹⁾ Particularly, it is observed a Stokes shift of 140 meV (30 meV) between the PL and OA peaks of PbS (PbSe) dots corresponding approximately to 12% (5%) of the recombination energy.

In order to perform the spatially resolved microluminescence, the samples were mounted in a variable temperature cryostat coupled with the measurement system. The idea lying behind this technique is to excite the sample surface using a tightly focused Ar^+ -ion laser beam (spot diameter averaging $3 \mu\text{m}$ wide). For PL measurements the emitted light was dispersed by a 0.5 m spectrometer, and the PL signal was synchronously detected by a nitrogen-cooled germanium detector. The energy transfer accounts for a broad luminescent region that surrounds the laser excitation spot. The magnified image of the luminescent region is scanned by the detection system attached to step motors and recorded as a function of the wavelength.⁴⁷⁾

To study energy transfer mechanisms, it is useful to compare both spectra (PL and OA) of Fig. 5, driving the attention to the Förster mechanism that is particularly the most important process that occurs at the nanometric scale.⁵⁰⁾ For an efficient energy transfer via Förster mechanism there should be interaction between transition dipoles of a donor and an acceptor to the spectral overlap of donor emission and acceptor absorption. So, a good overlap between the PL and OA curves would determine the efficiency of the energy transfer. The spectra of Fig. 5 confirm that this behaviour may be stronger in PbSe dots than in PbS dots. This is a reflection of a Stokes shift of about 5 times larger.

Figure 6 displays the PL spatial profile on the sample surface measured at different emission energies from the PL of the PbS nanocrystal QDs, at low temperature. The insert shows the corresponding PL spectrum. Taking into account the circular symmetry of the spatial profile around the laser spot, we just plot the PL intensity profile starting from the centre of the laser spot (set at $0 \mu\text{m}$). It is noted that the spatial profiles depend strongly on the detection energy. It is also found that as the detection energy decreases the PL intensity decay becomes larger. On the lowest detection energy the spatial decay can be as long as $200 \mu\text{m}$ (see curve “a” and the corresponding detection energy).

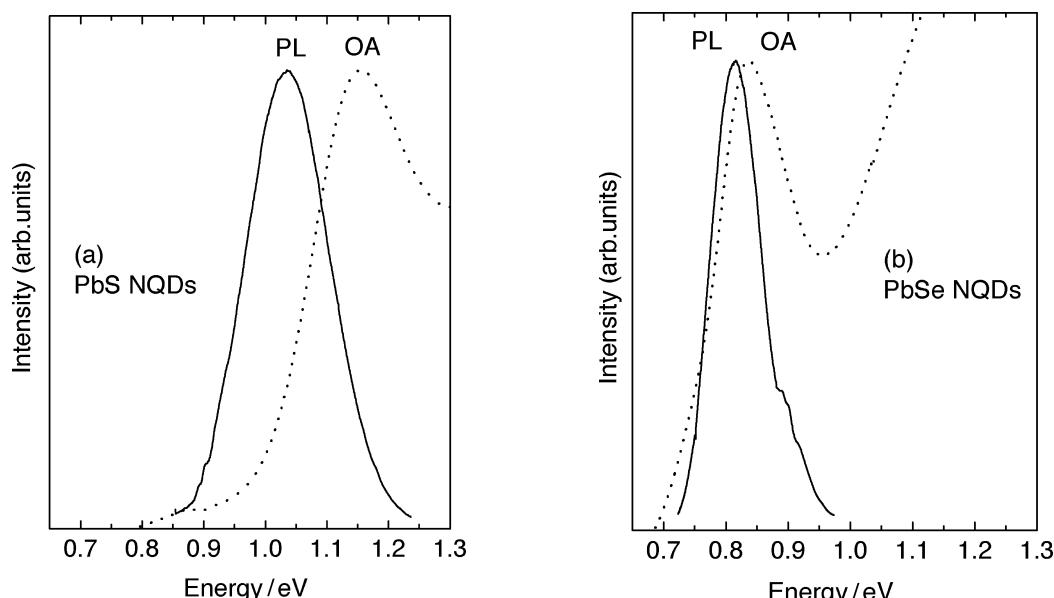


Fig. 5. Room temperature PL (solid lines) and optical absorption (dotted lines) spectra from PbS (left side) and PbSe (right side) nanocrystalline QDs.¹⁸⁾

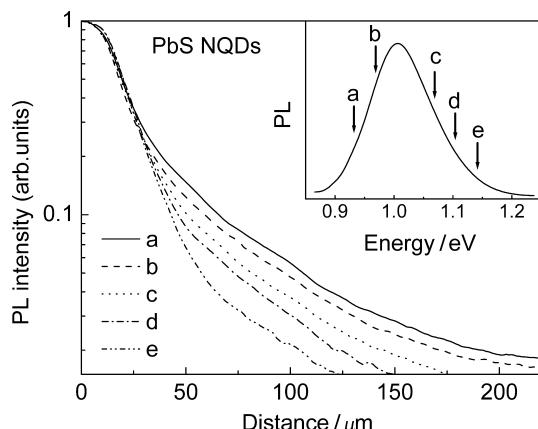


Fig. 6. PL spatial profiles measured at different detection energies obtained from PbS nanocrystal QDs. Inset shows the PL spectrum, and the vertical arrows indicate the detection energies that were used in PL spatial profile measurement.

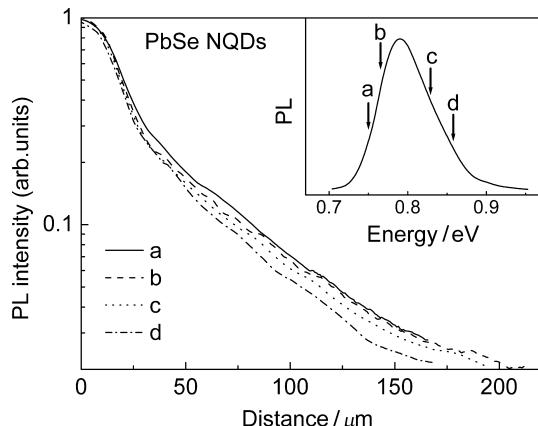


Fig. 7. PL spatial profile measured at different detection energies obtained from PbSe nanocrystal QDs. Inset shows the PL spectrum, and the vertical arrows indicate the detection energies that were used in PL spatial profile measurement.

What is happening is that the PL spatial profile widens steadily with increasing dot size (lower energies), been indicative of energy flowing into larger dots. These energy (size) dependent profiles unambiguously show direct energy transfer from small dots to larger dots. We argue that indirect coupling between different sub-bands via photon reabsorption plays an important role.

Figure 7 shows the results from similar measurements performed in Fig. 6, but for PbSe QDs. Our first comment is that the difference between the PL spatial profiles is smaller if compared to the PbS QDs. This behaviour was explained by using as argument the dispersion of quantum dot sizes in these two cases. From PL spectra, we find that the emission peak energy from PbS quantum dots is larger than that of PbSe QDs, using the same thermal treatment of the glass matrix. So, it implies that in our interesting range the sizes of PbS QDs are smaller compared to PbSe QDs. At the same time, the energy levels of a carrier in large QDs become less sensitive to the variation of QD size. Thus, it is expected that the difference between the PL spatial profiles becomes smaller, according to Fig. 7. However, in spite of small difference, the process of energy flow between different subsets of dots may also occur in PbSe dots in a reduced manner.

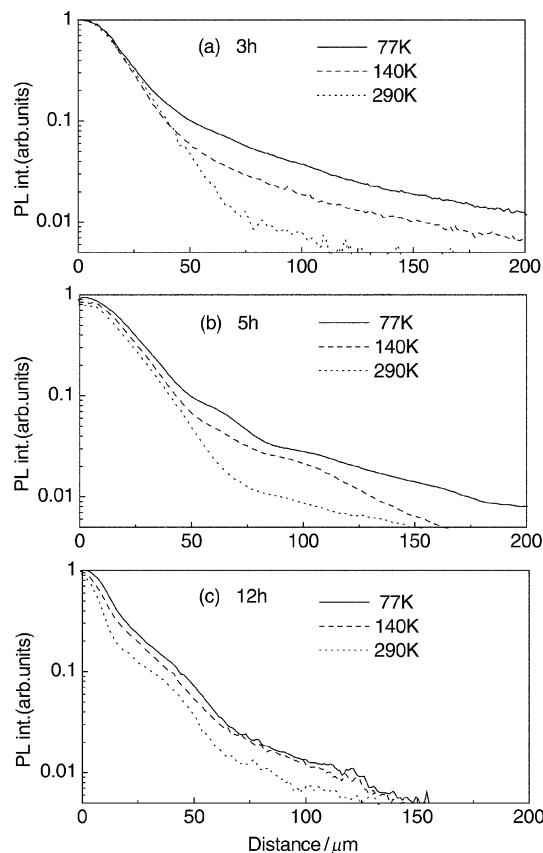


Fig. 8. PL spatial profile at different temperatures for different samples annealed at (a) 3 h, (b) 5 h and (c) 12 h. The PL peak position shifts to lower energies thus indicating that the QDs sizes increase as a function of the annealing time.⁴⁸⁾

The fact that the PbSe sample has a small variation with temperature accounts for the small distance between the energy levels implying lower interaction with the phonons in the matrix.⁴⁹⁾

Figure 8 displays the PL spatial profile on the sample surface obtained by detecting at the PL peak, at different temperatures. The samples were prepared with different annealing times of 3, 5 and 12 h. The thermal treatment clearly indicates that the QDs sizes increase as the annealing time increases. Thus, the PL peak position shifts to lower energies.⁴⁸⁾ Comparison between the data obtained from numerical calculation of the energy levels and the energy associated to the features observed in the optical absorption spectra can be used to estimate the PbS QD sizes.⁴⁹⁾ By doing this, the average PbS QD sizes for the 3, 5 and 12 h samples were estimated in 2.7, 4.0 and 9.0 nm, respectively. Remembering that the luminescence area on the surface is symmetrical around the laser excitation spot, the PL intensity profile is plotted from the centre of the laser spot located at 0 μm . By comparing the PL spatial profile for those samples, the 3 h sample clearly shows the widest profile, on the other hand, the 12 h sample shows the shortest profile. This picture demonstrates that the efficiency of energy transfer became higher for the sample with smaller dots (3 h sample), and lower for the sample with the largest dots (12 h sample).

The results demonstrate that the photon diffusion length that characterizes the PL spatial distribution is larger when we measure on the low-energy side of the PL spectrum. This behavior would account for the energy transfer between different subsets of dots. The subset of small dots that corresponds to the highest

emission energies works as a source of excitation for the subset of larger dots (lower energies). Moreover, photons that are emitted from the subset of small dot are strongly absorbed (by dots of same size or larger). On the other hand, photons that are emitted from the subset of large dots are only absorbed by the same subset. For this reason we would expect that the PL spatial distribution widens on the low-energy side of the PL spectrum.

It has been shown that efficient energy transfer occurs between quantum dots having a significant band gap energy difference, i.e., between dots of markedly different sizes.⁵⁰⁾ The measurements of the PbS dots indicate that due to the large Stoke shift reabsorption is less effective, and the energy transfer is more efficient between quantum dots having a significant band-gap energy difference. For this reason, varying the energy gap of the dots we find different spatial PL profiles. On the other hand, when we measure the PbSe nanocrystal QDs reabsorption is comparatively more effective, and the energy transfer may occur between dots having the same band-gap energy or small gap energy difference. Thus, this behaviour makes the PL profiles similar.

5. Conclusion

Compared to epitaxially grown techniques such as molecular beam epitaxy, semiconductor-doped glasses is low cost to fabricate. Because of their unique electronic and optical properties, semiconductor QDs found wide applications in the optoelectronics and optical communication. In photonics, they find applications such as saturable absorbers. PbS and PbSe QDs are interesting for infra-red application due to their narrow band gap. In summary, it was presented a novel technique to study energy transfer mechanisms using semiconductor nanocrystal doped glasses. The results directly revealed the energy transfer from smaller to larger dots in nanocrystal QDs. Energy transfer between different sizes dots can be quite reduced when the reabsorption is significantly high. Based on these findings, further improvements in size selectivity will permit highly efficient energy flows in nanocrystal doped glasses.

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