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Photoluminescence of CdSe nanocrystals embedded in a SiO_x thin film matrix

D. Nesheva[a,b], C. Raptis[b,*], Z. Levi[a], Z. Popovic[c], I. Hinic[c]

[a]*Institute of Solid State Physics, Bulgarian Academy of Sciences, 1784 Sofia, Bulgaria*
[b]*Department of Physics, National Technical University, Zografou Campus, GR-15780 Athens, Greece*
[c]*Institute of Physics, 11001 Belgrade, Yugoslavia*

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Abstract

Room- and low-temperature photoluminescence studies are reported on CdSe nanocrystals embedded in an SiO_x thin film matrix. The main spectral feature for all samples and both temperatures is a broad band whose position does not change considerably with nanocrystallite size. The band is assigned to recombination through defect states, whose energy depends on the nanocrystallite size in such a way that they counter-balance the similar dependence of the optical band gap on the nanocrystallite size. A noticeable asymmetry on the low-energy side of this band at low temperatures is attributed to the existence of a variety of surface defect states and/or a large number of volume defect states in these nanocrystals. There is evidence to suggest that the energy band diagram of CdSe nanocrystals in SiO_x matrix is considerably different from that of SiO_x/CdSe multi-quantum wells. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

The development of various techniques for growing nanocrystals (NCs) from a great variety of semiconductor materials (CdSe, CdS, CuBr, CuCl, Si, Ge, GaAs etc.) has opened the door for intensive studies of their linear, non-linear and electro-optical properties [1–4]. This enhanced interest is justified because NCs constitute intermediate systems between solids and isolated molecules, and because of their potential applications in optoelectronic devices.

The recombination processes taking place in semiconductors, and particularly in NCs, are of fundamental importance for their applications. The photoluminescence spectroscopic technique is widely used for the study of both radiative and non-radiative transitions of carriers in NCs. However, the spectra obtained from CdSe NCs embedded in various inorganic [2,5–9] or polymeric [3,10] matrices, as well as from NCs produced by means of the electroplating or chemical deposition techniques [11] are considerably different. The

* Corresponding author: Tel.: + 30-1-772-2926; fax: + 30-1-772-2928.

E-mail address: craptis@central.ntua.gr (C. Raptis)

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results are strongly influenced not only by the composition and structure of the matrix, but also by the preparation technique. The band-edge emission of CdSe NCs in a strongly confined regime has been generally attributed to electron transitions from the highest occupied to the lowest non-occupied molecular orbital [2,5,8–10]. Support for this interpretation has been given by calculations of the lowest excited states of CdSe NCs using various approximations [10,12,13]. A broad photoluminescence band has also been observed which is (red) shifted by 0.2 – 0.5 eV with respect to the optical band-gap position of NCs. It is generally connected with recombination via defect localized states in the gap [2,8,9].

In this article, photoluminescence studies are reported at room temperature and 10 K on CdSe NCs embedded in an SiO_x thin film matrix. A broad photoluminescence band, which peaks in the red region of the spectrum, is observed in all spectra measured. The influence of the nanocrystallite size on the intensity and shape of this band is discussed.

2. Experimental

2.1. Sample preparation

Thermal evaporation of powdered CdSe (Merk, "suprapure") and granular SiO from two independent tantalum crucibles was carried out at a vacuum of $\sim 10^{-3}$ Pa. During the deposition procedure, the substrates were maintained at room temperature and, when necessary, they were rotated at a rate varying between 8 and 30 turns/min. Film thickness and deposition rate of both materials were controlled during deposition by two preliminary calibrated quartz monitors MIKI-FFV. Their quartz crystal heads were fixed above the respective source and in order to ensure the same composition of the SiO_x matrix ($x \approx 1.5$) in all samples, the deposition rate of SiO_x was kept constant (~ 3.5 nm/s).

The technique used for the preparation of CdSe NCs in SiO_x thin film matrix has been described in detail previously [14]. By means of this technique CdSe nanoclusters are formed during deposition on Corning 7059 glass substrate. The average size of nanoclusters is approximately equal to the measured effective thickness of the island-type CdSe layers which alternate with 20 times thicker layers of SiO_x [14]. The CdSe nanoclusters obtained are crystallized by annealing the as-prepared films in air at 670 K for 90 min. It has been found [14] that CdSe NCs of hexagonal wurtzite structure are formed after annealing. The total effective thickness of CdSe deposited in these samples varies between 60 and 100 nm. The CdSe doping concentration of all samples prepared by this technique is ~ 7 wt%. The doping level has been estimated considering the total SiO_x and CdSe, thicknesses deposited during the preparation of a given layer and material densities of 2.1 and 3.1 g/cm^3 for SiO_x and CdSe, respectively.

2.2. Optical measurements

Photoluminescence spectra of CdSe-doped SiO_x thin films having various CdSe NC sizes (referred to as SiO_x–CdSe films from now on) were measured at room temperature and 10 K. The room-temperature spectra were excited by the 530.9 nm (2.315 eV) Kr^+ laser line (at a power density of 12 W/cm^2) and measured by a SPEX double monochromator in line with photon counting equipment. A cylindrical lens was used for the room-temperature measurements to avoid an undesirable heating of the samples. A closed-cycle He cryostat was used for the low-temperature measurements and the photoluminescence spectra were recorded by a similar system of a SPEX double monochromator and a photon counter. However, the 514.5 nm (2.41 eV) line of an Ar^+ laser was used at a power density of about 80 W/cm^2 for the excitation at 10 K. The difference in energy between the two laser lines used (514.5 and 530.9 nm) is approximately equal to the thermally induced decrease of the CdSe optical band gap with increasing temperature. In this situation, the increase of the optical band gap at low temperatures is counter-balanced by the higher (quantum) energy of the 514.5 nm line, thus the excitation of the luminescence spectra of NCs is similar in the two cases.

In order to determine the average nanocrystallite size d, X-ray diffraction measurements were carried

out using the Cu K_α line ($\lambda = 0.154$ nm) of an automated Siemens D-500 diffractometer. The formula $d = \lambda/\delta 2\Theta \cos\Theta$ [5,6] was employed where Θ and $\delta 2\Theta$ are the position and full-width at half-maximum (FWHM) of the [1 1 0] X-ray diffraction band of wurtzite CdSe. The values of d obtained should be considered only as indicative (particulary for NCs of small sizes) because of the large errors involved in this technique. Recent high-resolution electron-microscopy micrographs [15] of the films with NC size $d = 1.6$ and 2.5 nm (as determined by the X-ray diffraction data) have indicated average NC sizes about 60% and 50% greater than those obtained by X-ray measurements and a small size distribution. Since the former technique provides better accuracy than the latter, we may assume that the actual CdSe NC size in the films is higher than those quoted in the figures of this work. However, we maintain the nominal values of d for the NCs (as obtained by the X-ray data), because electron microscopy data are not available for all SiO_x–CdSe films of the series.

A small shift of the [1 1 0] peak to lower angles was observed in the X-ray spectrum with decreasing nanocrystallite size. This shift is probably due to a lattice contraction of CdSe NCs. The observed blue shift of the 1LO phonon band in the Raman scattering spectra of CdSe [16] and $CdS_{1-x}Se_x$ [17] NCs embedded in various glassy matrices has already been ascribed to such a lattice contraction. Unfortunately, because of the small total CdSe thickness in the films studied, the measured X-ray diffraction intensities were quite low and it was not possible to make a precise quantitative estimation of the size dependence of this contraction.

Room-temperature transmission spectra were taken with a two-beam spectrophotometer model SPECORD UV-VIS. In order to obtain the absorption spectra of SiO_x–CdSe films, photocurrent measurements were carried out in which planar Al or Ag contacts were used; in these experiments, the measured current–voltage characteristics were linear for applied fields of 10^2–10^3 V/cm. The photocurrent spectral dependence was measured when the samples were illuminated by a chopped (2 Hz) light beam from a tungsten-halogen source which passed through a single stage diffraction grating monochromator type MDR2 at a spectral resolution of up to 4 nm.

3. Results and Discussion

3.1. Room-temperature measurements

Room-temperature photoluminescence spectra of SiO_x–CdSe films with an average nanocrystallite size d of about 5.0, 3.0 and 1.6 nm (as determined by X-ray diffraction), are shown in Fig. 1. A broad band is seen in all spectra with some structure superimposed on it. Bearing in mind that the total thickness of these films is ~ 1.5 µm, one can assume that this structure is most likely due to light interference effects. Indeed, interference patterns were observed in the transmission spectra of these samples. The arrows in the spectrum of the film

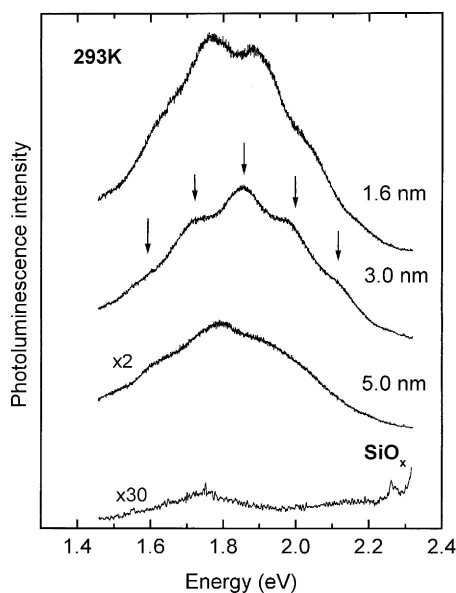

Fig. 1. Room-temperature photoluminescence spectra of CdSe NCs having average size of about 1.6, 3.0 and 5.0 nm produced by deposition in SiO_x thin film matrix. All spectra correspond to the same intensity scale. The arrows point to the positions of the interference maxima of the corresponding transmission spectra of the film. Samples were excited with the 530.9 nm Kr^+ laser line. A photoluminescence spectrum from a SiO_x single layer is also shown which is prepared and treated in a similar way as the SiO_x–CdSe films.

with NC size $d = 3$ nm (Fig. 1) point to the peak positions of the superimposed structures which coincide (in energy) with the interference maxima in the transmission spectrum of the same film. Hence, it appears that the room-temperature photoluminescence spectra of the SiO_x–CdSe films consist of just a structureless broad band. This band is satisfactorily fitted to a Gaussian (Fig. 2) having peak position at 1.82, 1.86 and 1.82 eV and FWHM of 0.52, 0.54 and 0.57 eV for the samples corresponding to $d = 1.6$, 3.0 and 5.0 nm, respectively.

Photocurrent measurements have been widely used in order to study absorption below and near the band edge of thin photoconductive films. The dark conductivity of the SiO_x–CdSe films varies between 10^{-10} and $10^{-8}\ \Omega^{-1}\mathrm{cm}^{-1}$ and the photocurrent to dark current ratio is between 10^2 and 10^3. This enabled us to determine the absorption coefficient α of the films with good accuracy, which cannot be done by combined transmission and reflection measurements on films having such a small (< 100 nm) CdSe total thickness. The α values (in arbitrary units) were obtained by employing both the approach used by Moddel et al. [18] and the constant photocurrent method developed by Vanecek and co-workers [19]. The absorption spectra of films having $d = 5.0$ and 3.0 nm are presented in Fig. 3a and 3b, respectively, along with the corresponding photoluminescence spectra. One can see that the first absorption maximum, corresponding to the optical band gap E_g of the sample, peaks at 1.92 eV for the film with $d = 5.0$ nm and shifts to 2.06 eV for the film with $d = 3.0$ nm. In general, the peak position of the first absorption maximum increases accordingly with decreasing d, as is demonstrated in Fig. 4 for films of various NC sizes. This result indicates that a size-induced increase of the optical band gap of CdSe NCs takes place. In Fig. 4 the systematic difference between the calculated (for finite wells of electrons and holes) and experimentally determined values of E_g is most likely due to the uncertainly in the NC size values (estimated from X-ray data) which were used for the former (see Section 2.2). However, the position of the photoluminescence band does not change considerably. The energy difference between the optical band gap and the

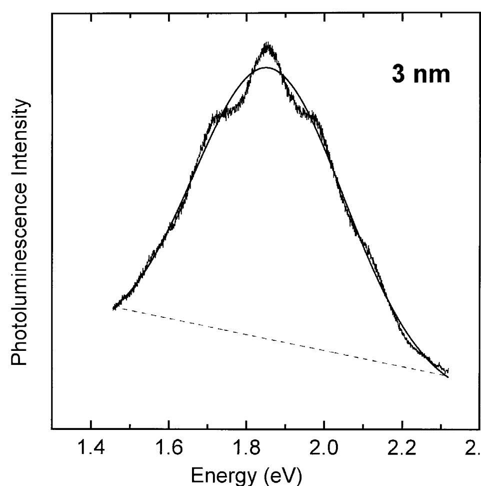

Fig. 2. A Gaussian fit of the photoluminescence band of an SiO_x–CdSe ($d = 3.0$ nm) film. The dashed line is the baseline taken into account when the peak position and FWHM of the band were determined.

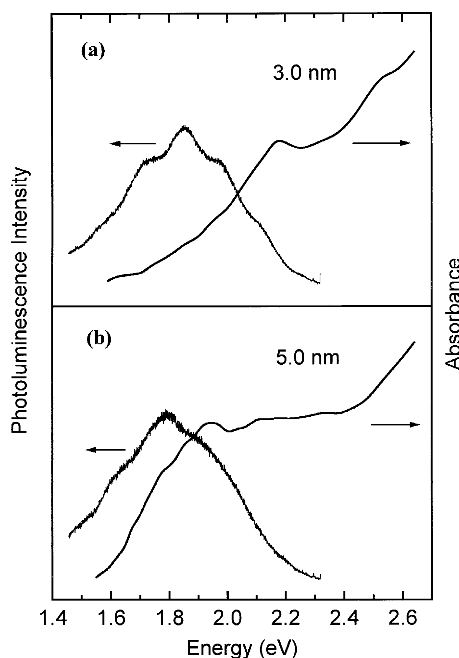

Fig. 3. Linear absorbance and photoluminescence of two SiO_x–CdSe films having average nanocrystallite size of about 3.0 (a) and 5.0 nm (b). It is clear that the optical band gap of the NCs increases with decreasing nanocrystallite size, but the photoluminescence band does not shift considerably.

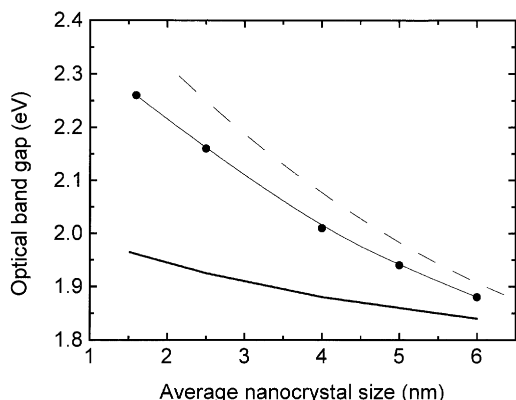

Fig. 4. Thickness dependencies of the minimum energy for electron transitions in CdSe NCs calculated [13] for either finite (0.6 eV) wells of electrons and holes (dashed line) or only an infinite well for holes (thick solid line) and the experimentally determined optical band gap of CdSe NCs embedded in SiO_x matrix (full circles). The line connecting the experimental points is a guide for the eye.

emission peak increases with decreasing d from about 0.15 eV to about 0.45 eV when the NC size changes from 5.0 to 1.6 nm. Moreover, the integrated intensity of photoluminescence increases with decreasing NC size, i.e. with increasing surface-to-volume ratio of the NCs (see Fig. 1).

As was mentioned above, two bands have been observed in the photoluminescence spectra of CdSe NCs in silicate glass [7,8,20] and GeO_2 thin film matrix [9]. The high-energy band in these spectra is slightly red shifted (< 100 meV) with respect to the first absorption peak. In most cases it is assigned to radiative transitions from the lowest level of the conduction band to the highest level of the valence band ($1S_e$–$1S_{3/2}$ transition [2,21]). The second band peaks at energies of 200–500 meV below the optical band-gap energy and its FWHM is about 400–500 meV [7–9]. It is considered that this band is due to recombination through defect states and its large FWHM is due to variations of the nanocrystallite size or to the existence of various kinds of surface or other defect states [8]. It was established that in NC doped glasses at room temperature [20], the intensity of the low-energy band is much higher than that of the band-edge photoluminescence. Moreover, the intensity of the high-energy band increases with decreasing temperature. This band shows a blue shift with decreasing nanocrystallite size, thus changing in accordance with the quantum-size increase of the optical band gap. As for the position of the low-energy band, it depends on several factors: the nanocrystallite size [8,9], the type of matrix employed and the method of NC preparation [2,3,9]. It is generally accepted that, in each NC, both recombination processes occur in a competitive manner. Alternatively, regarding the type of states involved in the two recombination processes, it has been suggested in a recent work [20] that the high-energy band originates from NCs in which the surface state concentration is rather low, while the low-energy one corresponds to NCs having a high level of surface defects.

Taking into account all the aforementioned characteristics of the photoluminescence bands of CdSe NCs, we conclude that the wide band observed at room temperature in SiO_x–CdSe films is predominantly caused by recombination through surface defect states. This conclusion is in agreement with the observed intensity increase of the band with decreasing NC size as well as with the large FWHM of the photoluminescence bands shown in Fig. 1. The absence of a high-energy band in the spectra indicates that the surface state recombination is the dominant process in all CdSe NCs of these films. Consequently, one may assume that the concentration of surface defects is high in all CdSe NCs. Because the optical non-linearity of $CdS_{1-x}Se_x$ doped glasses is proportionate to the surface defect concentration [20], there is even more interest in the study of the non-linear properties of the SiO_x–CdSe films.

The photoluminescence maximum does not shift appreciably when the average nanocrystallite size varies. It is different compared to the energy dependence of the respective maxima of other works [8,9] in which a blue shift of the low-energy band has been observed with decreasing NC size. According to a detailed model [8] of the surface state carrier recombination, it comprises two steps: at first, the photoexcited holes are captured by deep (strongly localized) acceptor surface states and then they recombine with free photoexcited electrons. It has been reported [8,9] that, if defect states are strongly localized, they may not experience the quantum-size effect intensely and, therefore, they

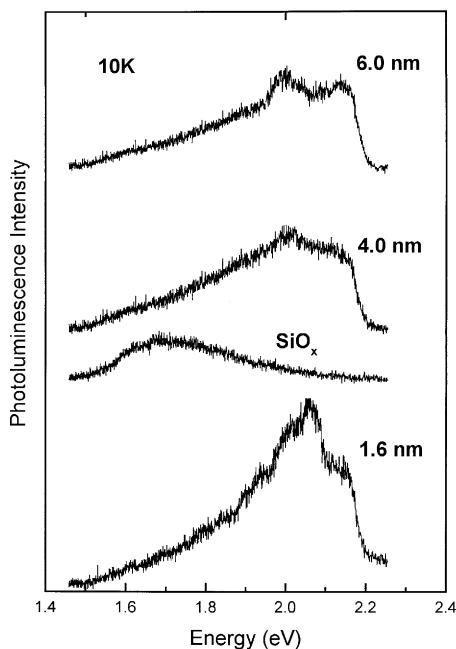

Fig. 5. Photoluminescence spectra taken at 10 K with the 514.5 nm Ar^+ laser line corresponding to CdSe NCs of various sizes in SiO_x thin film matrix. All spectra correspond to the same intensity scale. The photoluminescence spectrum of a SiO_x single layer is also shown for comparisons.

should not sustain a considerable change of their energy position. Consequently, the blue shift of the low-energy band observed [8,9] with decreasing CdSe NC size should be primarily related to a size-induced shift of the $1S_e$ level. Then, the more or less fixed position of the broad band (as determined by the fitting) observed in the photoluminescence spectra of SiO_x–CdSe films, may be explained in terms of a simultaneous increase in the energy of the defect states with decreasing NC radius [9,22]. In this situation, the energy distance between the bottom of the conduction band and the acceptor defect level remains approximately the same. Since the expected size-induced shift of the defect levels is not particularly strong, we may assume that the potential well for electrons in CdSe NCs should not be very deep. In order to verify this assumption we have compared (Fig. 4) the size dependence of the $1S_{3/2}$–$1S_e$ transition energy [13] (which has been calculated assuming either finite (0.6 eV) wells for electrons and holes or only one infinite well for holes in CdSe NCs) to the experimentally obtained size dependence of the optical band gap of the SiO_x–CdSe composite films. It is apparent from Fig. 4 that the experimental curve is situated closer to the upper theoretical curve. It indicates that there exist potential wells for both electrons and holes in CdSe NCs in SiO_x matrix and that they should be $\leqslant 0.6$ eV. This energy is lower than the value of 1.3 eV estimated from a band diagram of the potential well for holes in CdSe NCs of SiO_x/CdSe multi-quantum wells reported recently [23]. In this work, the size-induced increase in the optical band gap of CdSe NCs was calculated assuming a hole confinement only and was found in good agreement with the observed blue shift of the band-edge luminescence. All these imply that the band diagrams of the two systems (CdSe NCs in SiO_x–CdSe films and SiO_x/CdSe multi-quantum wells) are generally different and this should be attributed to the different dimensionality of both constituent materials of the systems, namely zero (CdSe) and three (SiO_x) in the films as compared to two (CdSe) and two (SiO_x) in the multi-quantum wells.

3.2. Low-temperature measurements

The photoluminescence spectra (excited by the 514.5 nm Ar^+ laser line) of SiO_x–CdSe films at $T = 10$ K are shown in Fig. 5 for three CdSe NC sizes ($d = 1.6, 4.0, 6.0$) along with the photoluminescence spectrum of a SiO_x single layer also at 10 K. A broad band positioned at 2.0–2.1 eV and having FWHM between 0.14 and 0.25 eV (as obtained by the fitting to Gaussian curves) appears in the spectra of films whose average NC size is between 1.6 and 6.0 nm. Unlike the respective band of the room-temperature spectra, the low-temperature band shows some structure. A shoulder at about 2.14 eV is also seen in the spectra of all samples. Besides, the main band, which peaks at 2.0–2.1 eV, is asymmetric on the low-energy side.

The shoulder at 2.14 eV may be related to photoluminescence from silicon nanoclusters having $d < 1$ nm (as has been calculated in Ref. [24]), which may have been formed in the SiO_x matrix ($x \approx 1.5$) [14] during the deposition and annealing procedures. The existence of Si nanoclusters has

already been assumed [23] on the basis of X-ray diffraction and transmission studies carried out in SiO_x–CdSe films and photoluminescence measurements in SiO_x/CdSe multi-quantum wells. Moreover, a superimposed Raman peak, which is shifted to about 500 cm^{-1} with reference to the 530.9 nm Kr^+ laser line, appears in the room-temperature photoluminescence spectra of the SiO_x single layer (see Fig. 1). The frequency of this Raman band is very close to the frequencies of the 1LO Raman band of Si NCs (504 cm^{-1}) and the broad Raman band of amorphous silicon (480 cm^{-1}) which have been observed in nanocrystalline Si/a-Si multilayers [25]. As in the room-temperature spectra, the maximum of the main band of the low-temperature photoluminescence of films having d between 1.6 and 4.0 nm is red shifted between 0.18 eV ($d = 4.0$ nm) and 0.35 eV ($d = 1.6$ nm) with respect to their experimentally determined (Fig. 4) optical band gap. The large red shift and FWHM (0.14–0.25 eV) of this band indicate that it is most likely caused by recombination through deep defect levels. Hence, under c.w. laser excitation, the defect level recombination process is dominant in all SiO_x–CdSe films having average NC size below 4 nm, even at very low temperatures. In a relevant work [9], under c.w laser excitation at $T = 5.5$ K, only defect radiative recombination was registered from small size ($d < 4.8$ nm) CdSe NCs in GeO_2 matrix. Some authors [2,26] have also detected at low temperature under c.w laser illumination band-edge photoluminescence of CdSe NCs having $d = 5$-6 nm, while other authors [8,9] have observed edge emission in NCs of smaller sizes only when the photoluminescence spectra are obtained via short-pulse laser excitation. Thus, the results of this study confirm the trends reported in the literature that the smaller the size of NCs embedded in a glass matrix, the stronger the effect of the interface detects on the NC photoluminescence.

The asymmetry on the low-energy side of the main photoluminescence band might be related to the existence of an appreciable number of CdSe NCs having sizes greater than the average size. However, as has been discussed above, in SiO_x–CdSe films, the position of the photoluminescence band owing to deep level recombination is not very sensitive to the NC size. Hence, the band asymmetry is most likely due to the existence of various kinds of defects in the CdSe NCs or, in the case of only one defect type, to an increasing defect energy with decreasing distance of the defect from the NC centre [9,22].

Finally, we would like to comment on the fact that the FWHM (0.14–0.25 eV, as obtained by the fittings) of the main band in the low-temperature spectra is at least two times smaller than that (0.52–0.57 eV) of the same band at room temperature. Also the FWHM of the band due to surface level recombination of CdSe NCs in GeO_2 matrix [9] and $CdS_{1-x}Se_x$ NCs in Corning 2.59 and 2.61 filters [27], is approximately the same over wide temperature ranges (5.5–290 K [9] and 6–90 K [27]). A possible reason for this might be a temperature-dependent pressure effect of the SiO_x matrix on CdSe NCs owing to different temperature dependence of the thermal expansion coefficients of the two materials. This assumption needs verification through a more detailed study.

4. Conclusions

NCs of CdSe embedded in an SiO_x thin film matrix have been produced and photoluminescence studies have been carried out at room temperature and 10 K in order to study the effects of nanocrystallite size on the position and concentration of the defect states of CdSe NCs. A structureless broad band has been observed in the room-temperature spectra of CdSe NCs having average diameter $d \leqslant 5$ nm. It has been ascribed to recombination through surface defect states, whose energy depends on the nanocrystallite size. Under c.w. laser excitation, the radiative recombination through defect states is dominant in all samples and at both temperatures. Based on this result, it is concluded that a great concentration of surface defects exists in CdSe NCs embedded in SiO_x thin film matrix. A strong asymmetry on the low-energy side of the defect photoluminescence band has been observed at 10 K in the spectrum of the CdSe NCs. This result is related to a variety of surface defects or/and a high concentration of volume defects existing in these NCs. The high defect concentration observed makes SiO_x–CdSe films interesting

candidates for non-linear optical applications. Comparing the observed size-induced increase of the optical band gap of CdSe NCs with the calculated one [13] and bearing in mind the size-independent position of the room-temperature photoluminescence band, we have estimated that the wells for electrons and holes in CdSe NCs in SiO_x matrix are about 0.6 eV. Thus, it is implied that the energy band diagrams of SiO_x–CdSe films and SiO_x/CdSe multi-quantum well structures are essentially different and this is attributed to the variety of dimensionality among the constituent materials.

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