Transfer mechanisms in semiconductor hybrids with colloidal core/shell quantum dots on ZnSe substrates

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Abstract

Hybrid systems consisting of colloidal CdS/ZnS core/shell quantum dots on ZnSe semiconductor substrates have been studied by continuous-wave and nanosecond time-resolved photoluminescence. On the basis of kinetic calculations, we studied the interplay between the possible transfer processes in these hybrids. The considered transfer mechanisms were resonance energy transfer, photon reabsorption, electron and hole tunneling. Depending on the size of the CdS cores the dominating transfer mechanism is changing. Carrier tunneling was found only for quantum dots in direct contact to the substrate. For large quantum dots a hole tunneling was found, whereas in case of small dots the fast electron tunneling is decisive. Eventually, we were able to determine the conduction band offset between CdS and ZnSe to 0.56 eV at 10 K.

Keywords: time-resolved photoluminescence, semiconductor hybrids, colloidal CdS quantum dots, ZnSe, carrier tunneling, energy transfers

(Some figures may appear in colour only in the online journal)

1. Introduction

Colloidal quantum dots (QDs) are attractive building blocks for functionalization of semiconductors in electronic and optoelectronic devices. The wet chemical synthesis of colloidal QDs and the solution based preparation of hybrids consisting of colloidal QDs and semiconductor substrates allow for low cost devices with a huge variety of physical properties that can be modified by size, shape and composition of the QDs as well as by the interaction with the substrate. Promising examples are e.g. the use of colloidal QDs in light emitting diodes [1–3], which might enable the next generation of displays [4], high efficient QD solar cells [5], field-effect transistors [6] or spintronic devices [7]. Since the first report on a colloidal quantum dot solar cell in 2005 [8] the power-conversion has been increased considerably. Gi-Hwan Kim et al and Xinzheng Lan, et al reported power conversion efficiencies of more than 10.5% [9, 10].

In optoelectronic devices basically two different transfer mechanisms could be used in accordance to the aimed application which are either carrier transfer or energy transfer. The most decisive parameter is the band alignment between the colloidal QD and the substrate. It is well known, that for an efficient carrier transfer the acceptor-energy level should be well below the excited donor state. An efficient energy transfer will take place if the donor excitation energy is substantially higher than the acceptor excitation.

In this paper we used a model system to carefully study the interplay between electron- and hole-tunneling and energy transfer in hybrids consisting of colloidal QDs and semiconductor substrates. The precise knowledge about the dominating process in dependence on the respective conduction and valence band offsets is important for novel applications. To
reveal the dominating processes, we studied the QD exciton dynamics by analyzing the emission decay in the framework of kinetic model calculations. For our studies we used CdS/ZnS core/shell QDs. It is well known that a thin ZnS shell provides an efficient passivation of the surface trap states, giving rise to a strongly enhanced luminescence quantum yield in the excitonic region. Energy loss due to non-radiative recombination or recombination via in-gap states can be suppressed substantially [11]. This is a fundamental prerequisite for the use of QDs in applications, which rely on high quantum efficiencies. The tunneling and energy transfer is of course somewhat hindered by the ZnS shell, but the gain obtained upon surface passivation clearly predominates.

2. Experimental

2.1 Methods

We performed photoluminescence (PL) measurements by means of a standard setup with a high-resolution grating spectrometer. For the steady-state PL, a He-Cd laser with 325 nm (3.8 eV) was used. The time resolved photoluminescence measurements were carried out with a pulsed Nd:YAG laser operating at a wavelength of 355 nm. The repetition rate was 10 Hz and the pulse had a temporal width of 3 ns. The laser was focused on the sample. The sample was put in a cryostat and cooled down to 10 K. The PL was analyzed with a 250 nm spectrometer using a 300 lines per millimeter grating which was blazed for a wavelength of 500 nm. The resulting spectra were recorded with a gated iStar intensified charged coupled device (ICCD). The minimum gate width used was 6 ns, it was gradually increased to measure the spectra at late times after excitation, up to 250 ns for the spectra 1 µs after excitation. The complete temporal behavior of the PL was obtained by recording the PL spectrum for different times after the laser excitation.

The absorption measurements were performed by means of an Agilent 8453 UV–VIS spectrophotometer.

2.2 Samples

Core/shell-type CdS/ZnS QDs stabilized with oleic acid (see figure 1a) and dissolved in toluene were purchased from Sigma-Aldrich, with a stated emission wavelength of 400 and 450 nm. The difference in the emission wavelength is caused by the confinement effect due to the different size of the QDs. The QDs with a stated emission wavelength of 400 nm are referred to as small QDs in this paper, the QDs with emission wavelength of 450 nm are referred to as large QDs.

The QDs have been transferred to quartz substrates via blade coating to prepare the QD-semiconductor hybrids. Blade coating is a known method to deposit quantum dot layers [12, 13], that can also be used for large scale deposition. A drop from the solution of the QDs dissolved in toluene was placed on the substrate and a blade was used to remove excess material, resulting in an almost uniform distribution of the QDs on the substrate (thin layers). Thicker layers with an uneven thickness were prepared by depositing a known amount of the solution and let it dry without removing any material from the sample.

For comparison quartz, ZnSe or ZnMnSe layers grown by molecular beam epitaxy (MBE) on GaAs substrates have been used as substrates. One type of substrate has a 100 nm ZnSe film on top, whereas the other type has a 20 nm thick ZnMnSe layer with a manganese concentration of 16% on top and an undoped ZnSe buffer layer underneath. Throughout the paper, these samples are simply referred to as ZnSe and ZnMnSe.

The resulting QD layers were characterized by absorption measurements using layers of small QDs on quartz (see figure 3). The absorbance signal becomes weaker when the QDs have been transferred to the quartz substrate in comparison to QDs in solution due to the relatively low number of measured QDs. To estimate the thickness of the thin layer, its absorbance is compared to the absorbance of a thick layer with an estimated amount of QDs. We come back to this point later. The characteristic absorption peak at around 390 nm is not visible in case of the thin layer, but from the absorbance in the UV-region, the thickness of the thin layer can be estimated to be roughly a tenth of the thick layer. Multiple measurements of separately prepared thin layers show only little differences.

The core diameter D of the CdS/ZnS QDs can be estimated from the spectral position of the absorbance peak by using the empirical function [14]:

\[
D(nm) = (−6.65Z1 * 10^{-8}) * \lambda^3 + (1.95Z5 * 10^{-4}) * \lambda^2 \\
- (9.23Z5 * 10^{-2}) * \lambda + 13.29
\]  

(1)

This results in an average CdS core diameter of 3.1 nm for the small QDs. Alternatively, the size can be calculated from the lowest transition energy using the Brus equation (2)
The energy of the QD $E_{QD}$ has been determined by taking the mean of the spectral position of the emission and absorption maximum. All the other data needed for the calculation are taken from [17]. The band gap energy at room temperature of bulk CdS is $E_{\text{gap}} = 2.42$ eV, the effective masses for the electron and hole are $m^*_e = 0.21$ and $m^*_h = 0.8$ and the relative permittivity is 8.9. This results in a core diameter for the small QDs of $D = 2R = 3.2$ nm in almost perfect agreement with the afore determined value.

$$E_{QD} = E_{\text{gap}} + \frac{\hbar^2}{8R^2} \left( \frac{1}{m^*_e} + \frac{1}{m^*_h} \right) - \frac{1.8e^2}{4\pi\varepsilon_0 \varepsilon_r R} \quad (2)$$

Likewise the core diameter of the larger QDs is estimated to be $D = 4.8$ nm using Yu’s formula or $D = 4.4$ nm with Brus equation.

We now want to estimate the thickness of the layers. The average full core/shell QD diameter of the small QDs is about 4.5 nm according to the manufacturer. The difference to the above calculated 3.1 nm is due the ZnS shell. The particle concentration of the small QDs in solution is calculated from the given mass concentration and the mass of a single QD (estimated from its size) to be $5 \times 10^{15}$ QDs/ml. The thick QD layer is formed by an evenly spread dried 10 µl drop on a 1 cm$^2$ quartz plate. The average thickness of the thick layer would then be at minimum about 10 monolayers of QDs assuming a cubic arrangement with a lattice parameter of 4.5 nm, e.g. no extra space for the organic shell or at maximum 20 monolayers of QDs assuming about 2 nm extra distance between the ZnS/CdS QDs due to the organic shell. It is most interesting to note at this point, that according to the absorption measurements the thin QD-layers, showing only a tenth of the absorption, are almost perfect with one or two monolayers of QDs only.

3. Results and discussion

In figure 3 the typical photoluminescence (PL) spectra are shown for the ZnSe and ZnMnSe substrates and for the two types of QDs on quartz measured at $T = 10$ K. It can be seen that the PL-maximum of the small QDs at $E_{QD} = 3.13$ eV is energetically well above the ZnSe exciton peak at 2.796 eV. The strong quantum confinement effect of the CdS-QDs allows therefore for radiationless resonance energy transfer to the ZnSe layer, which is prohibited in bulk material due to the smaller bandgap of CdS compered to ZnSe (see figure 4). It is known, however, that CdS and ZnSe heterostructures exhibit a type-II band alignment. This allows for a hole tunneling from CdS to ZnSe in case of bulk material. It is noteworthy at this point, that the excitonic transition in ZnMnSe with 16% is shifted to higher energy compared to ZnSe, as also reported earlier in [18–20].

In figure 5 the temperature dependence of the PL intensity of thin QD-layers on ZnSe substrate is compared to the temperature dependence on quartz substrate at different temperatures. An increase of the QD PL intensity on quartz substrate
can be seen with rising temperature starting around 200 K. This intensity increase can be explained by thermal activation of electrons from trap states, as discussed already in references [21, 22]. The thermal release acts as feeding process for the bright excitons. The origin of the feeding states has been discussed in various papers, being either the triplet exciton state in the QDs [23, 24] or disorder induced trap states at the interface causing the well known blinking effect [25, 26]. In case of our core/shell QDs the blinking is negligible and interface states should therefore play a minor role. The thermal excitation of the triplet state should be the overwhelming process. It is interesting to note, that for the QD-layers on ZnSe, this increase of the PL intensity is not observed. This is typical behavior if carrier or energy transfer takes place from the QDs to the substrate.

To reveal the transfer mechanism from the QDs to ZnSe we studied the recombination dynamics of the CdS/ZnS-QD singlet states on different substrates with and without transfer processes. Figure 6 shows the PL decay curves of a thin layer of small QDs on quartz substrate measured at different temperatures. It should be mentioned at this point that the value of 8–10 eV are well above our QD emission and we can assume that no energy transfer and no carrier loss from the QDs to the quartz substrate will take place. The CdS/ZnS-QD decay at 10 K (blue triangles in figure 6) exhibits clearly a non-exponential transient. This is due to fact that the PL is caused by an ensemble of QDs with varying diameters and decay times. The size distribution of the QDs also causes the broadening of the PL linewidth [27, 28], whereas the contribution of in-gap states observed in alloyed QDs [29] plays only a minor role in our core/shell QDs. As can be seen in figure 3 the PL-halfwidths is about 180 meV which corresponds to a core diameter variation between 2.8 and 3.5 nm, what is in good agreement with a diameter variation of ±10% according to the manufacturer. Bi-exponential or stretched exponential functions are often used to describe the non-exponential transients [30–33]. In this paper we assume a distribution of different decay times for the QD ensemble, assuming a single exponential decay for each QD. The total intensity $I_{tot}$ of the ensemble is then given by equation (3).

$$I_{tot}(t) = \sum_{\tau'} I_{\tau'}(t)$$

(3)

$I_{\tau'}(t)$ are the intensities of the individual QDs with decay times $\tau'$. Going towards a continuous distribution of decay times, the time dependence of the total intensity is then given by equation (4) with $\rho(\tau') = \frac{I_{\tau'}(0)}{\hat{I}_0}$ reflecting the initial intensity distribution.

$$I_{tot}(t) = \hat{I}_0 \int_0^\infty \rho(\tau') \exp(-\frac{t}{\tau'}) d\tau'$$

(4)

The exact distribution is not known, but in this work it will be approximated by a log-normal distribution (see equation (5)), with $\tau_m$ being the median time of the distribution and $\sigma$ is the standard deviation on the log-scale. The use of a log-normal distribution for the decay rates of nanoparticles was suggested earlier by van Driel et al [34]. It is worth mentioning, that recently a measured size distribution of colloidal QDs was well approximated by a log-normal distribution [35].

$$\rho(\tau') = \frac{1}{\tau' \sigma \sqrt{2\pi}} \exp\left(\frac{-(\ln \tau' - \ln \tau_m)^2}{2\sigma^2}\right)$$

(5)

The full blue line depicted in figure 6 is the best fit using equation (4) for the experimental curve measured at 10 K with a median of $\tau_m = 22$ ns and $\sigma = 1.1$. With increasing temperature up to 70 K the decay curves get faster. The respective median values are $\tau_m = 15.5$ ns for $T = 30$ K and $\tau_m = 8$ ns.
for $T = 70$ K. The faster decay with increasing temperature is a typical behavior and caused by an increasing electron-phonon-coupling promoting the transfer processes but also enhance nonradiative losses. It is interesting to note, that at room temperature the PL-decay is much slower and exhibits a pronounced long living part due to the feeding process from the triplet states discussed above. Considering this feeding process in the kinetic model yield an exponential term with a substantially enhanced time constant $\tau_{\text{trf}}$. This decay time is determined by the triplet lifetime and the feeding probability of the singlet states. To keep the kinetic model as simple as possible we included the same exponential term for the feeding process of all QDs and determined the respective time by fitting. The transient of the ensemble PL is then given by equation (6) with $\tau_{\text{trf}}$, being the lifetime of the dark triplet states acting as quasi feeding time. The best fit in figure 6 at $T = 290$ K yields a median decay time of 9 ns and a feeding time of $\tau_{\text{trf}} = 260$ ns.

$$I(t) = A_0 \int_0^\infty \rho(\tau') \exp\left(\frac{-t}{\tau'}\right) d\tau' + B_0 \exp\left(\frac{-t}{\tau_{\text{trf}}}\right)$$

As next step we want to include the transfer process from the QD-layer to the substrate. We have to extend our kinetic model now by including a transfer time $\tau_{\text{trf}}$ to account for the energy transfer or carrier tunneling. It is important to note, that only those QDs take part in the transfer process which are close enough to the interface. For this reason a factor $c = N_g/N_{\text{total}}$ is introduced representing the proportion of QDs $N_g$ at the interface which can lose their energy or carriers to the substrate. As aforesaid, the feeding of the singlets from the triplets is not observable for QDs at the interface with strong transfer. Including all this, the resulting intensity decay is then given by equation (7).

$$I(t) = c \cdot I^{\text{trf}}(t) + (1-c) \cdot I^{\text{tot}}(t) = (1-c + c \cdot \exp\left(\frac{-t}{\tau_{\text{trf}}}\right))$$

$$\times A_0 \int_0^\infty \rho(\tau') \exp\left(\frac{-t}{\tau'}\right) d\tau' + (1-c) \cdot B_0 \exp\left(\frac{-t}{\tau_{\text{trf}}}\right)$$

In figure 7 the PL transient of a thin and thick layer of the small QDs on ZnSe is shown in comparison with a QD layer on quartz. Only a slightly faster transient is observable for the thick layer on ZnSe in comparison to the layer on quartz. A much faster decay can be seen, however, for the thin layer on ZnSe. This clearly indicates a transfer process from the QDs to the ZnSe substrate. We can conclude qualitatively that only QDs placed close enough to the interface lose their energy or carriers. In case of the thick layer the overwhelming amount of QDs exhibit the usual radiative lifetimes without any transfer process to the ZnSe substrate.

A more quantitative discussion is possible by fitting the experimental curves with equation (7). The mean transfer time of the additional loss process can be deduced straightforwardly by fitting the experimental curves of the thin and thick layer on ZnSe. All the parameters beside $\tau_{\text{trf}}$ and $c$ are taken from the QD-layer on quartz at 10 K where no transfer to the substrate is observable. The resulting transfer time for both layers is $\tau_{\text{trf}} = 8.9$ ns at 10 K, but the proportion factor is strongly different. For the thin layers $c = 0.91, \text{e.g.}$ more than 90% of the QDs take part in the transfer process, whereas for the thick layer $c = 0.32$ only about one third take part. It is worth mentioning, that the transfer to the ZnSe substrate is similarly effective at room temperature (see the inset of figure 7). The fit gives $c = 0.60$ and $\tau_{\text{trf}} = 6.6$ ns, i.e. a slightly faster transfer time at room temperature compared to 10 K but a reduced $c$, which is caused by an enhanced radiationless recombination due to the stronger electron–phonon coupling at room temperature.

We now come to the question, which transfer process is responsible for the fast decay. As already mentioned, energy

Figure 7. Time-resolved PL decay curves of layers of small QDs on quartz and ZnSe substrates at 10 K (inset at room temperature), taken at 400 nm. Full lines are calculated (see text for details).

Figure 8. Time-resolved PL decay curves of the exciton from the ZnSe substrate without a QD layer on top (blue squares) and with a thin (green dots) or a thick layer (red triangles) of small QDs on top, taken at 443 nm.
transfer is possible, which is either Förster-like resonance energy transfer (FRET) or a tunneling of excitons as entity [36, 37]. The band alignment basically allows for hole transfer. However, as can be seen in figure 4 the strong quantum confinement calculated by means of equation (2) allows also for electron tunneling. We come back to this point later.

To reveal the underlying transfer processes, we examined the photoluminescence decay of the substrate. In figure 8 the PL transients are depicted for the ZnSe excitons from the substrate without QDs (red triangles) and with a thin QD-layer (green dots). It is obvious that the transients are the same. In case of an energy transfer from the QDs to the ZnSe exciton, the ZnSe decay time would be slower. From this we can conclude that there is no indication for such an energy transfer. A bi-exponential fit yields $\tau_1 = 7.2$ ns and $\tau_2 = 50$ ns. The blue curve in figure 8 depicts the decay of the ZnSe substrate exciton PL with a thick layer of QDs on top of the substrate. The slower decay is a clear indication for energy gain from the QDs. This gain is, however, simply due to reabsorption of QD-PL. We know from earlier investigations, that the radiationless transfer is relevant only at very small spatial distances between donor and acceptor [21]. For large distances, the reabsorption always dominates. The full blue line is due to the ZnSe decay plus reabsorbed photons. At later times, the ZnSe decay resembles the QD decay. We conclude from these results, that FRET or exciton tunneling does not play a prominent role in this hybrid system.

This conclusion is strongly supported by the measurement of a thin QD-layer on ZnMnSe depicted in figure 9. The decay curve of the small QDs on ZnSe and quartz are depicted additionally for comparison. Obviously, the transfer is substantially suppressed on the ZnMnSe substrate. Using equation (7) for the fit (red curve in figure 9) we get a much smaller value for $c$ assuming the transfer time is not changed ($c = 0.37$, $\tau_{\text{trf}} = 8.9$ns). Only 37% of the QDs take part in the transfer on ZnMnSe compared to 91% on ZnSe.

It is important to note at this point, that the bandgap energy $E_{\text{gZnMnSe}}$ of ZnMnSe with $x_{\text{Mn}} = 0.16$ is only about 30 meV higher than the bandgap of ZnSe. Energy transfer from the small QDs to the exciton states of the substrate would still be possible. The energy transfer from the singlet QD-states to the localized Mn states need not to be considered, since it is strongly suppressed due to the spin selection rule [38]. However, hole tunneling would still be possible since the large valence band offset is only slightly changed. In reference [39] it was shown, that the conduction band of ZnMnSe shifts to higher energies for $x_{\text{Mn}} = 0.16$ by about 10–25 meV compared to ZnSe. Based on these results, it can be concluded that the electron transfer is the dominating mechanism for the small QDs. The strong quantum confinement of the electron states in case of the small QDs (see figure 4) obviously enables the electron tunneling, which is already decisively hampered by the CB shift towards higher energies in case of the ZnMnSe substrate. This strong dependence is expected only, if QD electron states are almost in resonance to CB of ZnMnSe. The calculated confinement effect for the electrons using equation (2) leads to a CB shift of 0.75 eV. We have to take into account, however, also that the coulomb binding between the electron and hole must be released to transfer the electron away from the hole. The coulomb binding energy calculated with equation (2) is about 190 meV. Based on these, we have to conclude that the CB offset between bulk CdS without confinement and ZnSe is about 0.56 eV. It should be noted at this point, that there are different offset values in the literature. Theoretical values are 630 meV as a result of a calculated valence band offset of 350 meV [40] or about 600 meV determined by means of the universal alignment of the hydrogen levels in semiconductors based on ab initio calculations [41]. Experimental values reported so far were 800 meV [42] and 670 meV [43].

Finally, we want to verify our interpretation using the large QDs. As can be seen in figure 3 the QD recombination energy is below the ZnSe and ZnMnSe excitons. Therefore, we do not expect energy transfer. In figure 10 the decay curves of thin layers with large QDs ($\lambda_{\text{em}} = 450$ nm) on ZnSe and ZnMnSe are compared with the decay on quartz at $T = 10$ K. The decay curve on quartz is fitted according to equation (4) with a median time of 7 ns and a $\sigma = 1.3$. The faster decay of the larger QDs was discussed in [44] and explained by the different magnitude of the energy splitting between the bright and dark exciton states. The decay is slightly faster on ZnSe and ZnMnSe. The fitting with equation (7) yield a transfer time of $\tau_{\text{trf}} = 10$ns in both cases and a value of $c = 0.42$ for ZnSe and $c = 0.54$ for ZnMnSe. The shift of the CB due to quantum confinement is less than 0.4 eV for the large QDs and the Coulomb binding energy is about 120 meV. Therefore, the electron transfer can be excluded. There is only one transfer channel left to explain the transients of the large QDs ZnSe and ZnMnSe compared to quartz, namely the hole transfer. The large QDs thus exhibit a similar decay on ZnSe and ZnMnSe.
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Figure 10. Time-resolved PL decay curves of layers of large QDs on quartz, ZnSe and ZnMnSe substrates at 10 K, taken at 450 nm. Full lines are calculated (see text for details).

4. Summary

In summary, we studied hybrid systems consisting of CdS/ZnS core/shell QDs on ZnSe substrates and were able to reveal different transfer processes from the QDs to the substrate. In dependence on the size and band gap energy of the QDs the dominating transfer mechanism is changed. A detailed analysis is possible at 10 K. If the singlet transition energy of the QDs is smaller than the ZnSe band gap a hole tunneling was found. If the singlet energy of the QDs exceeds the ZnSe bandgap a Förster-like energy transfer is basically possible but does not play a significant role compared to reabsorption in case of thick QD layers. For small enough QDs the confinement shift of the electron states is larger than the ZnSe bandgap a Förster-like energy transfer is basically possible but does not play a significant role compared to reabsorption in case of thick QD layers. For small enough QDs close to the ZnSe surface transfer their excited electrons. At room temperature the transfer time is little faster with 8.9 ns at 10 K and more than 90% of the QDs close to the ZnSe surface transfer their excited electrons. In that case a very efficient electron transfer from the QDs to the substrate is found. The tunneling time is 8.9 ns at 10 K and more than 90% of the QDs close to the ZnSe surface transfer their excited electrons. At room temperature the transfer time is little faster with 6.6 ns and the total amount of transferred electrons is reduced to about 60%. This reduction is mainly due to the enhanced nonradiative recombination in the dots caused by the increased electron phonon coupling at room temperature. Nonetheless, these values found are still promising for feasible applications. Eventually, we were able to determine the bulk conduction band offset between CdS and ZnSe to be about 0.56 eV.

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