# Photoluminescence of Colloidal CdSe/ZnS Quantum Dots Under Oxygen Atmosphere

Gia-Wei Shu, Wan-Zhen Lee, I.-Jen Shu, Ji-Lin Shen, James Cheng-An Lin, Walter H. Chang, Ruoh-Chyu Ruaan, and Wu Ching Chou

Abstract—The effects of oxygen versus vacuum ambients on colloidal CdSe/ZnS quantum dots (QDs) were studied using both continuous and time-resolved photoluminescence (PL) measurements. The PL intensities were found to be an order of magnitude higher in an oxygen atmosphere, which is explained by the passivation of surface defects by oxygen absorption. The decay of PL intensities can be best fitted by a biexponential function with lifetimes of approximately 1 ns for the fast decay and approximately 10 ns for the slow decay. Based on the emission-energy dependence of carrier lifetimes and of the amplitude ratio of the fast-decay component to the slow-decay component, we suggest that the fast and slow PL decay of colloidal CdSe/ZnS QDs is caused by the recombination of delocalized carriers in the internal core states and the localized carriers in the surface states, respectively.

Index Terms—Optical properties, quantum dots (QDs).

## I. INTRODUCTION

MICONDUCTOR quantum dots (QDs) that are smaller than the bulk exciton Bohr radius provide a nearly zero-dimensional system in which carriers are confined in all spatial directions. The quantum confinement results in discrete electronic levels, which can be tuned by varying the QD size. The optical properties of the semiconductor QDs are expected to exhibit a high luminescence yield and thermal stability because the QDs have a  $\delta$ -like density of states and strong quantum confinement. Their novel optical properties cause semiconductor QDs to have many applications in opto-electronic devices such as optical switches, light-emitting diodes (LEDs), and lasers [1], [2]. In recent years, colloidal semiconductor QDs have been chemically coupled to biomolecules such as peptides, DNA, and proteins [3]–[7]. These QD bioconjugates can be used as biomedical fluorescence labels for investigating biomolecular interactions and developing highly sensitive detection and imaging [3]–[7]. Although colloidal semiconductor QDs are promising materials in numerous applications, the origin of their luminescence remains

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the topic of some controversy. The near-edge photoluminescence (PL) in colloidal semiconductor QDs has been attributed to the recombination of the carriers from surface states [8]–[11]. This is generally acceptable since the heterostructure interfaces, unsaturated bonds, and adsorbed foreign species on the surface create surface states with energies within the bandgap of the material. The surface states may act to localize or trap the photogenerated electrons and/or holes on the surface. However, some experiments have revealed that the near-edge PL originates from the internal core states in QDs—not the surface states [12]–[14]. The enhanced electron-hole exchange interaction and strong confinement in the core states indicates that the spin degeneracy of the band-edge exciton is lifted and a dark exciton state is formed. The presence of a dark exciton can explain the PL properties in QDs. Therefore, the origin of PL in colloidal QDs remains an open question.

This study elucidates PL in colloidal CdSe/ZnS QDs in an atmosphere of oxygen. Introducing the oxygen to QDs can enhance their PL intensity by an order of magnitude without causing photo-oxidation. Time-resolved PL decay measurements provide a complementary method for studying the PL properties of QDs, using the kinetics of electron-hole recombination as a probe. The PL decay of QDs was monitored at various emission wavelengths and in an atmosphere of oxygen, and we suggest that the fast and slow decay of PL may originate from the recombination of the internal core states and the localized surface states, respectively.

## II. EXPERIMENTAL DETAILS

The investigated colloidal core/shell CdSe/ZnS QDs were synthesized by the following procedure. 0.30 g of cadmium oxide (CdO), 1.30 g of tretradecylphosphonic acid (TDPA), and 25.0 g of tri-n-octylphosphine oxide (TOPO) were loaded into a 250-mL flask and heated to 320 °C under argon flow. After the CdO was totally dissolved in TDPA and TOPO, the solution was cooled to 300 °C and 4.45 mL of selenium stock solution (0.5 M of selenium solution in tributylphosphine, TOP) was injected. 1.5 mL of precursor solution (made by mixing a 1.75 mL of ZnMe<sub>2</sub> (2.0 M in toluene) and S(Si(CH<sub>3</sub>))<sub>2</sub> in TBP) was then added dropwise into the mixture to cover a layer of ZnS. 20 mg of TOP/TOPO capped CdSe/ZnS QDs was mixed with a 200-mg 11-mercaptoundecanoic acid (MUA) in a reaction vessel to synthesize the carboxylated QDs. Methanol was added and the pH value was adjusted to about ten with tetramethylammonium hydroxide. The mixture was heated under reflux for 2 h. After it was cooled to room temperature,

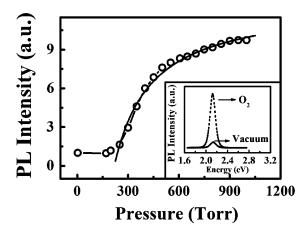


Fig. 1. PL intensity of CdSe/ZnS QDs under different oxygen pressures. The solid line displays the result calculated using (1). The PL signals in a vacuum environment (solid line) and an oxygen atmosphere (dashed line) are shown in the inset.

the resulting water-soluble QDs were precipitated with tetrahydrofuran and separated by centrifugation.

The PL measurements were obtained at room temperature using a pulsed GaN diode laser operated at a wavelength of 396 nm as the excitation source. The diode laser produced light pulses with a duration of 50 ps and a repetition rate of 1 MHz. The collected luminescence was directly projected into a grating spectrometer and then detected with a high-speed photomultiplier tube (PMT). PL decay signals were measured using the technique of time-correlated single-photon counting (TCSPC) and a PC plug-in time-correlated counting card. The overall temporal response function of the system was 300 ps.

### III. RESULTS AND DISCUSSION

The solid line in the inset in Fig. 1 displays the PL signal of colloidal CdSe/ZnS QDs in a vacuum environment (at a pressure of  $10^{-3}$  torr). The PL peak energy was centered at 2.13 eV, which is assigned to the near bandgap transition. No PL was observed from the deep trap states at energies of lower than 2.13 eV, indicating that the CdSe/ZnS QDs have good optical qualities. The dashed line in the inset of Fig. 1 shows the PL signal of CdSe/ZnS QDs in an atmosphere of oxygen at a pressure of 1 atm. The PL intensity in the oxygen atmosphere was enhanced by an order of magnitude greater than that in a vacuum. The significant role of oxygen in the fluorescence enhancement from single CdSe/ZnS QDs has also been recently demonstrated [15]. The open circles in Fig. 1 present the PL intensity of CdSe/ZnS QDs at various oxygen pressures. The PL intensity remains unchanged in the low-pressure range  $(10^{-3}-200 \text{ torr})$ , increasing after 200 torr. The influence of oxygen pressure on PL intensity can be described by the quantity of the monomolecular adsorption layer and the pressure, as formulated by Zhao et al. [16] and Langmuir [17] as follows:

$$I(P) = A \frac{K \times P}{1 + K \times P} \tag{1}$$

where I(P) is the PL intensity, P is the gas pressure, and A and K are constants [16]. The solid line in Fig. 1 plots the results calculated using (1). A good fit to experiment is found for

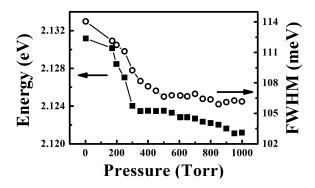


Fig. 2. PL peak energy (closed squares) and FWHM (open circles) of CdSe/ZnS QDs as a function of oxygen pressure.

the pressure range of P>200 torr. In the low-pressure range, the deviation between calculations and experiments may arise from the decrease in the amount of oxygen absorbed on the surface of QDs, originating from the trapping of oxygen by MUA molecules. The good fit in Fig. 1 reveals that the enhancement of PL intensity is related to the adsorption of the first monolayer of oxygen on the surface of QDs.

Fig. 2 displays the PL full-width at half maximum (FWHM) of CdSe/ZnS QDs as a function of oxygen pressure, indicating that the FWHM decreases as the oxygen pressure increases. The enhanced PL intensity and reduced PL FWHM in the oxygen atmosphere suggest that the surface traps of QDs are passivated by introducing oxygen. Surface defects such as Se or/and Cd dangling bonds can introduce surface trap states with energies that are within the bandgap of the material. The adsorbed oxygen may interact with surface defects, which are directly involved in the nonradiative recombination of the photogenerated carriers [18]–[20]. For instance, Se dangling bonds can be passivated by introducing oxygen, reducing the main trap sites for holes [21]. If the surface defects are eliminated by oxygen, then the contribution from radiative recombination should increase, enhancing PL intensity. The reaction of oxygen with the surface defects of CdSe QDs can be of two types—chemisorbed interaction and physisorbed interaction. The chemisorbed interaction involves the surface oxidation of CdSe, whereby oxygen is irreversibly chemisored onto the surface defects, forming a surface oxide. The oxidation product, which can result in smaller QDs and a loss in PL efficiency, has been suggested to be  $SeO_2$  or  $CdSeO_x$ (x = 2 or 3) [22]. The other one is a weakly interaction, whereby oxygen is physisorbed onto the CdSe surface. The physisorbed interaction can be eliminated by the evacuation of oxygen. PL experiments were performed after the evacuation of oxygen to identify the mechanism of oxygen passivation. The PL properties (intensity, peak position, and FWHM) in the vacuum recovered their initial values, which are consistent with the results in the inset of Fig. 1. Moreover, refilling with oxygen recovered the PL enhancement, indicating that the PL enhancement is a reversible effect and the oxygen passivation of surface defects should be attributed to the physisorbed interaction. The PL enhancement from single CdSe/ZnS QDs upon sudden exposure to air has been studied very recently [15]. Actual chemical modification, such as ligand reaction, has been suggested not to take place at the surface of ODs.

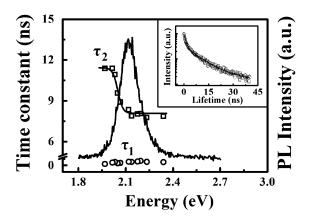


Fig. 3. Mission energy dependence of  $\tau_1$  (open circles) and  $\tau_2$  (open squares). The solid line displays the calculated  $\tau_2$  using (3). The spectrum of CdSe/ZnS QDs is also shown. PL decay profile of CdSe/ZnS QDs in a vacuum environment is shown in the inset. The solid line in the inset displays the calculated result using (2).

Fig. 2 also shows the PL peak position of CdSe/ZnS QDs as a function of oxygen pressure. The peak energy decreases as the oxygen pressure increases. Oxygen has been reported to be able to interact with CdSe under light illumination, producing evaporating oxides [8], [21]-[23]. This photooxidation of QD reduces the effective core diameter of QDs to decrease and causes the PL peak to undergo a blue shift. The red shift of the PL peak in Fig. 2 indicates that no photoxidation occurred in the sample. The red shift of the PL peak can be also explained by the passivation of surface defects. Oxygen had already eliminated the nonradiative surface defects so the carriers had more opportunities to recombine radiatively on the surface. The radiative recombination on the QD surface can occur at some energy potential minima because of the carrier localization (as will be demonstrated later) [24], leading to a red shift of the PL peak. Additionally, the electronic confinement effect may contribute to the PL red shift. Oxygen, which is very electronegative, can adsorb onto the surface of QDs via a transfer of electrons from the semiconductor to the oxygen [18]. Accordingly, the electron wave function confined in the CdSe core can extend toward the ZnS shell layer as oxygen adsorbs on QDs. In an analogous effect, the core diameter of QDs electronically increases, causing the PL peak to be red-shifted.

The inset of Fig. 3 displays the normalized PL decay profile of CdSe/ZnS QDs in a vacuum environment. The decay can be well fitted by a biexponential function

$$I(t) = A_1 e^{-\frac{t}{\tau_1}} + A_2 e^{-\frac{t}{\tau_2}} \tag{2}$$

where  $\tau_1(\tau_2)$  represents the fast (slow) decay time, and  $A_1(A_2)$  represents the amplitude of the fast-decay (slow-decay) component at t=0. The solid line in the inset of Fig. 3 shows the fitted result, which is in good agreement with the experimental results. The PL decay mechanism of CdSe/ZnS QDs can be further analyzed by the emission energy dependence of the lifetimes. The open squares and circles in Fig. 3 show the measured lifetimes of slow and fast decay, respectively, for different emission energies. As shown in Fig. 3, the lifetimes of slow PL decay fall as the emission energy increases, the relationship of which is

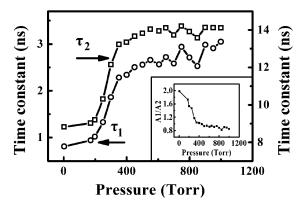


Fig. 4.  $au_1$  (open circles) and  $au_2$  (open squares) as a function of oxygen pressure. The inset shows the PL amplitude ratio  $A_1/A_2$  as a function of oxygen pressure.

characteristic of the localization effect [25]. Localization associated with the structural disorder or surface states may generate localized states with different energies. Accordingly, localized carriers can be transferred from higher energy sites to lower energy sites through a relaxation process. The decay rate of localized carriers is expressed as the radiative recombination rate plus the relaxation rate to lower energy sites. Thus, the decay time decreases as the emission energy increases, as displayed in Fig. 3. Assuming the density of localized tail states is proportional to  $\exp(-E/E_0)$ , the emission energy dependence of  $\tau_2$  can be fitted with the function [26]

$$\tau(E) = \frac{\tau_{\rm rad}}{1 + \exp[(E - E_{\rm me})/E_0]}$$
(3)

where  $\tau_{\rm rad}$  is the radiative lifetime,  $E_{\rm me}$  is the energy similar to the mobility edge, and  $E_0$  is a characteristic energy that represents the depth of the tail states.  $\tau_2$ , described by (3), is plotted as a solid line in Fig. 3. A good fit with experiment confirms the effect of carrier localization.

The PL amplitude ratio of the fast-decay component  $A_1$  to the slow-decay component  $A_2$  was analyzed to explore further the origins of the fast and slow PL decays. The inset of Fig. 4 plots the PL amplitude ratio  $A_1/A_2$  as a function of oxygen pressure, indicating that the PL amplitude ratio  $A_1/A_2$  decreases (the slow-decay component increases) as the oxygen pressure increases. Accordingly, the slow PL decay should involve the surface-related PL since its lifetime increases with the pressure of oxygen, which is adsorbed onto the surface of QDs. Additionally, the amplitude of the slow-decay component increases while the PL intensity is enhanced (as shown in Fig. 1), revealing that the slow-decay component is related to the radiative recombination. The results in Fig. 3 and the inset in Fig. 4 suggest that the slow-decay component of PL in (2) originates from the recombination of carriers in the *surface localized states*. However,  $\tau_1$  is independent of emission energy (as shown in Fig. 3) and shorter than  $\tau_2$ , which is associated with the surface states, thus,  $\tau_1$  is suggested to involve the recombination of delocalized carriers in the internal core states. Fig. 4 plots the lifetimes of the PL decay of CdSe/ZnS QDs as a function of oxygen pressure. Both  $\tau_1$  and  $\tau_2$  increase with increasing the oxygen pressure. The removal of the surface traps by introducing oxygen reduces the nonradiative recombination and enables the carriers on the surface to survive longer, a fact which accounts for the increase of  $\tau_2$  with the oxygen pressure. The increase of  $\tau_1$  with an increase in the oxygen pressure can be explained by the electronic confinement effect. As the electronic confinement decreases due to oxygen adsorption, the wave-function overlap between electrons and holes is reduced accordingly, leading to an increase of the carrier lifetimes in the core states.

# IV. CONCLUSION

In summary, the PL of colloidal CdSe/ZnS QDs in an oxygen atmosphere was studied using PL methods. The intensity of PL increased by an order of magnitude as the pressure of oxygen increased from vacuum to 1 atm. This enhancement is explained by the passivation of the surface defects by oxygen. Based on the emission energy dependence of the lifetimes and the PL amplitude ratio  $A_1/A_2$ , we suggest that the fast and slow PL decay of colloidal CdSe/ZnS QDs involves the recombination of the delocalized carriers in the internal core states and the localized carriers in the surface states, respectively.

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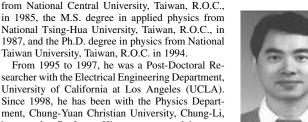


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