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DOI

[10.1016/j.jlumin.2015.07.029](https://doi.org/10.1016/j.jlumin.2015.07.029)

Publication date

2015

Document Version

Final published version

Published in

Journal of Luminescence

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[Link to publication](#)

Citation for published version (APA):

De Angelis, R., Casalboni, M., De Matteis, F., Hatami, F., Masselink, W. T., Zhang, H., & Proposito, P. (2015). Chemical sensitivity of InP/In_{0.48}Ga_{0.52}P surface quantum dots studied by time-resolved photoluminescence spectroscopy. *Journal of Luminescence*, 168, 54-58. <https://doi.org/10.1016/j.jlumin.2015.07.029>

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Journal of Luminescence

journal homepage: www.elsevier.com/locate/jlumin

Chemical sensitivity of InP/In_{0.48}Ga_{0.52}P surface quantum dots studied by time-resolved photoluminescence spectroscopy

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ARTICLE INFO

Article history:

Received 2 April 2015

Received in revised form

2 July 2015

Accepted 21 July 2015

Available online 1 August 2015

Keywords:

Vapour sensor

Quantum dot

Semiconductor nanocrystal

InP

Photoluminescence

ABSTRACT

InP/InGaP surface quantum dots represent an attractive material for optical chemical sensors since they show a remarkable near infra-red emission at room temperature, whose intensity increases rapidly and reversibly depending on the composition of the environmental atmosphere. We show here their emission properties by time resolved photoluminescence spectroscopy investigation. Photoluminescence transients with and without chemical solvent vapours (methanol, chlorophorm, acetone and water) were fitted with a 3-exponential decay law with times of about 0.5 ns, 2 ns and 7 ns. The measurements revealed a weak effect on chlorophorm, acetone and water, while the initial decay time of InP surface quantum dots increases (up to 15%) upon methanol vapour exposure, indicating that the organic molecules efficiently saturate QD non-radiative surface states.

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1. Introduction

The photophysical properties of semiconductor quantum dots (QDs), such as their broad absorption band and size-dependant spectral emission, together with the high effective surface area available for interaction with target chemicals are attractive for applications like chem-/bio-sensors and lab-on-chips [1–3]. So far, most of the reports of optical chemical sensing with QDs are based on colloidal quantum dots (CQDs) synthesized by wet chemistry methods [4–6].

Recently, a new approach has been introduced which is based on Surface QDs (SQDs) grown by epitaxy on solid substrates [7,8]. Usually, epitaxial quantum dots require a capping layer of a wider band gap semiconductor providing charge carriers confinement and non-radiative surface state passivation. Such structures are called Buried QDs (BQDs) and the direct contact with the external environment (usually air) is avoided, differently with respect to the uncapped SQDs. However, in some relevant cases it is possible to achieve sufficient quantum confinement and radiative emission in SQDs, thanks to air acting as an effective potential barrier and surface states saturation [9–12]. SQDs present the advantage of possible short-range interaction between dots and environment allowing their exploitation as sensor (e.g. for organic molecules).

A strong sensitivity to the surface environment has been reported for SQDs made of InAs [13], InGaAs [7] and InP [8]. These QDs are not suitable to be used in solution, and thus cannot be exploited for application like *in vivo* imaging and diagnostics. However, the peculiarity that they are directly grown on a conventional wafer represents a major advantage for chem-/bio-sensor and lab-on-chip design since the integration of CQDs into semiconductor devices is still a challenging task.

On this regard, InP/InGaP SQDs have some key advantages. Their emission intensity is only 2–5 times lower than that of InP/InGaP BQDs [9], which is quite outstanding compared to analogous systems where the intensity for uncapped QDs is usually orders of magnitude lower than for capped ones [14]. Moreover, their near infra-red (NIR) emission spans from 1.47 eV to 1.65 eV, thus matching fibre optics and biological tissue transparency windows. Therefore, InP/InGaP SQDs are suitable for a wide range of application.

In our previous works, we characterized the methanol sensitivity of InP/InGaP SQDs by continuous wave photoluminescence (CW-PL) spectroscopy [8,15–18]. We observed a rapid and reversible increase in the PL intensity, proportional to the methanol concentration. A model of the sensing mechanism would be beneficial in order to fine tuning the property of the dots and the whole semiconductor heterostructure to achieve methanol sensing with resolution suitable for practical application.

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Here, we report on the sensing mechanism investigated by time resolved photoluminescence (TR-PL) spectroscopy. We compare the response of the InP SQDs to nitrogen and methanol, water, acetone and chloroform solvents. We found that the decay transients of the PL in nitrogen environment can be well fitted by a three exponential decay function having characteristic times of 0.5, 2.0 and 7.0 ns. When the gas environment is changed introducing fixed amount of solvent vapour an increase of the fast and intermediate times was observed. Such an effect is particularly evident for methanol where a 15% enhancement of the fast decay time has been measured. The results are fully compatible with a model we recently suggested [8,16,18] based on the saturation effect of the QD non-radiative surface states by the vapour organic molecules. The model suggested is here experimentally confirmed for the first time while the alternative explanation of an off-set of the air potential barrier due to the adsorption of chemicals is ruled out.

2. Experimental

2.1. Synthesis and morphological characterization

Self-assembled epitaxial InP/In_{0.48}Ga_{0.52}P SQDs were grown by gas source molecular beam epitaxy (GS-MBE) in a RIBER 21-T MBE system on (100) GaAs substrate. The sample synthesis follows a well-established procedure [19–24] that allows dot density control [24]. After oxide desorption, a 100-nm thick GaAs buffer was grown at 550 °C at a rate of 0.4 atomic monolayer per second (ML/s), followed by 250 nm In_{0.48}Ga_{0.52}P grown at 460 °C. Then, the substrate temperature was lowered to about 410 °C and the InP was deposited with a rate of 0.85 ML/s, resulting in the formation of the quantum dots, followed by cooling down the sample to 20 °C. SQDs formation is induced by the 3.8% lattice mismatch between In_{0.48}Ga_{0.52}P and InP according to the Stranski–Krastanow growth mechanism. The growth process has been monitored *in situ* with high-energy electron diffraction (RHEED) technique, allowing for the observation of the 2 × 1-reconstruction of the In_{0.48}Ga_{0.52}P surface. *Ex-situ* compositional analyses have been accomplished by means of double-crystal X-ray diffractometry (DXCD). Samples present lattice-matched In_{0.48}Ga_{0.52}P to GaAs with high crystal quality. Structural characterization has been accomplished with atomic force microscopy (AFM) using an *ex-situ* Nanoscope IIIa in the tapping mode to image the surfaces of the sample.

2.2. Photoluminescence and vapour sensing measurements

Continuous wave photoluminescence (CW-PL) was measured in static atmosphere at room temperature (25 °C) and atmosphere pressure (760 Torr) using the 450 nm line of a semiconductor diode laser with power density of 90 mW/cm² for the excitation. PL was collected and analysed by a 25 cm monochromator (ARC SpectraPro-300i) and a photomultiplier (Hamamatsu R636-10) with lock-in detection technique. Photoluminescence spectra and decay times were recorded using a confocal microscope setup exploiting an inverted light microscope (Olympus IX71) equipped with an oil immersion 100 × objective mounted on a piezo scanning stage. Excitation light source was a Ti:Sapphire laser Chameleon ULTRA-II (Coherent 980 nm, 80 fs, 80 MHz repetition rate) equipped with an OPO (optical parametric oscillator (Coherent) tuned at 488 nm and a pulse peaker to reduce the repetition rate. Excitation light was coupled into the adapted confocal unit via a polarization maintaining monomode fibre and filtered using an excitation filter. An appropriate dichroic mirror and specific bandpass optical filters were used to separate the fluorescence from the excitation light. The emitted light was then passed through a 80/20 beam splitter and sent to a single photon avalanche diode (Perkin-Elmer,

SPCM-AQR-13) connected to the PCI-board for Time-Correlated Single Photon Counting (PicoQuant, TimeHarp200) for the lifetimes measurements and to a spectrograph (Acton Research Corp., Spectra Pro-150) equipped with a CCD camera (Roper Scientific, PhotonMax 512B) for the fluorescence surface images. Measurements were performed under the same conditions (760 Torr and 25 °C) in a sealed flow cell with an optical window where the atmosphere can be changed from pure N₂ to a mixture of solvent vapour and N₂. Such mixtures were produced by bubbling the liquid solvent in a Drechsel bottle to obtain the saturated vapour of the solvent and mixing it with N₂ in a controlled amount. The gas mixture was injected into the cell by means of a graded syringe. The TR-PL measurements were taken in static atmosphere. Then, the initial condition was restored flowing pure N₂ for 5 min. The liquid solvents used to produce the vapours were analytical reagent grade methanol (MeOH) 99.8%, chloroform (CF) 99.8% and acetone (Ac) 99.5%. All of them were anhydrous chemicals by Sigma-Aldrich, used without further purification. The saturated vapour pressure was calculated according to the Antoine's law with equation parameters taken from NIST database and mixture of a fixed concentration was prepared. The solvent concentration in the mixture has been estimated with an accuracy of 5%.

3. Results and discussion

The investigated sample is homogeneously covered by lens shaped SQDs with a lateral size of about 20–30 nm and a height of 2–4 nm. The dot density is 9×10^{10} dots/cm². Fig. 1 shows a 3D atomic force microscopy (AFM) micrograph of the sample.

The continuous wave photoluminescence (CW-PL) spectrum of InP SQDs shown in Fig. 2 presents a broadband emission with peak position at 1.47 eV and full width at half maximum (FWHM) of 98 meV. The emission energy and bandsape depend on dot size, size distribution and dot density. In a recent paper, we discussed the origin of InP/InGaP SQDs wide emission with an asymmetric profile presenting k.p bandstructure and optical calculations. We modelled InP SQDs starting from real structures from atomic force microscopy images, showing that the holes energy levels and the oscillator strengths are strongly dependant on the actual shape of the dots and are different from ideal structures such as lens or truncated pyramidal shapes [25]. Moreover, the experimental optical spectrum of one of our samples has been related to a numerical ensemble average derived from single dot calculations, with dot sizes and size distribution extrapolated from AFM

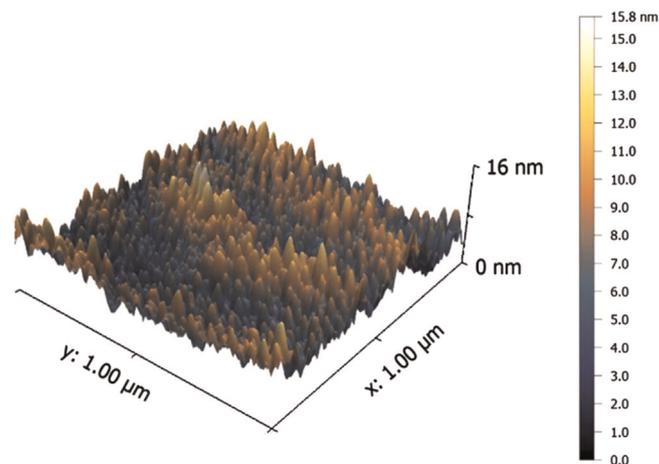


Fig. 1. $1 \times 1 \mu\text{m}^2$ 3D AFM micrograph of InP SQDs. The sample exhibits lens shaped SQDs with lateral size and height of 20–30 nm and 2–4 nm, respectively. The dot density is 9×10^{10} dots/cm².

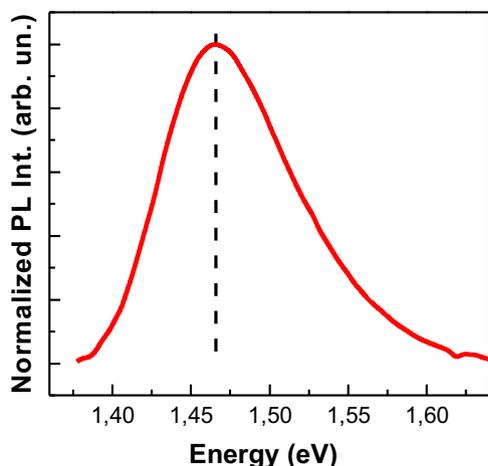


Fig. 2. Room temperature (25 °C) CW-PL spectrum of InP/InGaP SQDs corresponding to the sample showed in Fig. 1. The measurement was carried out with excitation density of 90 mW/cm² (450 nm laser) in nitrogen atmosphere.

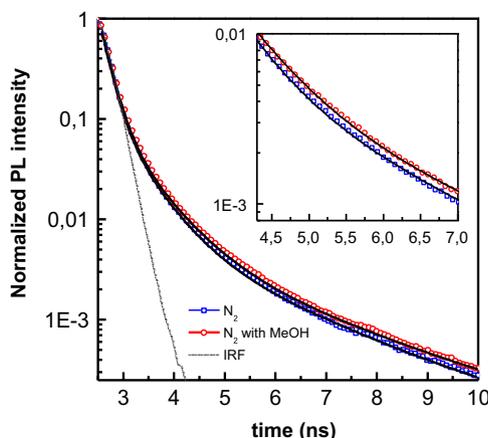


Fig. 3. PL transients from InP/InGaP SQDs with (red circles) and without (blue squares) methanol vapour (2×10^4 ppm) at 25 °C. Transients were recorded at the peak energy maximum. Black lines show 3-exponential decay fits and grey dashed line shows the IRF. The inset show a zoom from 4.5 to 7 ns. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

micrographs [25]. Recently, we also showed that a molecular contribute due to the lateral coupling between the dots in such high-density samples must be taken into account in order to have a strong similarity with the experimental spectrum [26].

In previous studies [8,15–18] we investigated the chemical sensitivity of InP/InGaP SQDs. We found that the composition of the surrounding gaseous environment strongly affects their PL intensity. In particular, we observed a significant PL enhancement with methanol vapour. Such findings pointed out the need of a model of the sensing mechanism involving the role of surface states acting as non-radiative centres. Our hypothesis was that methanol molecules saturate surface trap-states, effectively hindering non-radiative recombination of the photogenerated carriers. The correctness of such model remained as an open question in our previous works.

In order to ascertain the origin of the enhancement of the luminescence intensity when the sample of InP/InGaP SQDs is in a solvent vapour atmosphere we performed TR-PL measurement with and without vapours. Fig. 3 shows the PL transients from InP/InGaP SQDs, measured at 1.47 eV (the peak of the emission spectrum) in N₂ and methanol, and its initial response function (IRF). Taking into account the IRF, the transients (with and without

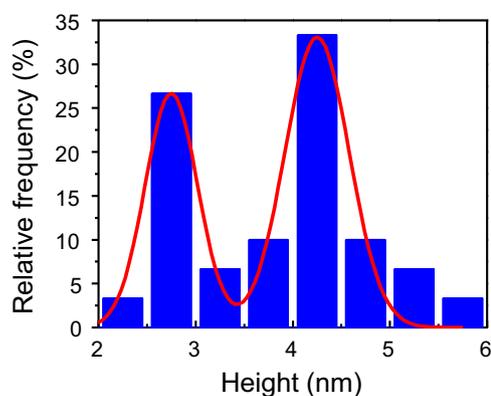


Fig. 4. Histogram of the dot height from AFM micrograph statistical analysis (blue bars), and fit of the data (red line).

methanol) can be well fitted with a 3-exponential decay law with an initial fast (τ_1), an intermediate (τ_2) and a slower (τ_3) decay time of about 0.5 ns, 2 ns and 7 ns, respectively.

The origin of a multi-exponential behaviour of surface InP QDs has been discussed in detail by Hestroffer et al., in comparison with InP BQDs [9]. While BQDs showed a single exponential behaviour, the transient from uncapped SQDs showed a multi-exponential behaviour. In the paper by Hestroffer et al. a 2-exponential decay law with times of 2 and 8 ns at 90 K was found. The initial decay time is similar to that of capped QDs, indicating comparable oscillator strengths (consistent with similar PL intensity). In the investigated sample we found a 0.5 ns and a 2 ns components, which are due to the emission of confined states in QDs. The two different times may arise from the different size of the dots. Smaller dots due to a higher quantum confinement are expected to exhibit a higher emission energy and a shorter radiative decay time. From the structural analysis of our sample it is possible to observe that the dot height present a bimodal Gaussian distribution, with peak positions of 2.8 and 4.3 nm (Fig. 4). Such figure has been obtained with a statistical analysis of the size and the shape of about 30 individual dots measured from a 600 nm \times 600 nm AFM micrograph. For InP SQDs with bigger lateral size compared to their height, one of the major parameters which is responsible for the emission energy and thus radiative lifetime is the dot height. Likely, the presence of two different radiative lifetimes is a consequence of such bimodal height distribution in this particular sample.

The longest component (7 ns) can be attributed either to coupling between confined states in QDs and surface states or to coupling between QDs themselves. In particular, a recent theoretical investigation by Baretin et al. showed the relevance of InP SQDs lateral coupling in determining emission energy and optical spectra. According to this study, the appearance of molecular terms in the optical spectra are likely to occur and are expected in our samples [26]. The experimental evidence of the presence of such a long component in the PL transients further reinforces such theory.

When the sample is exposed to methanol vapour the initial and middle components, τ_1 and τ_2 , of the PL transient lengthen while for τ_3 we did not observe any significant change. In order to test the sensitivity and selectivity of the InP SQDs we performed a series of analogous experiments with other common solvents, namely chloroform, acetone and water. The results are summarized in table 1.

From these data we evaluated the lifetime relative change induced by the presence of the vapour solvents as $\Delta\tau_\alpha/\tau_\alpha\% = (\tau_{\text{solvent}} - \tau_{\text{N}_2})/\tau_{\text{N}_2}\%$, where $\alpha = 1, 2, 3$ indicate the fast, intermediate and long decay times, respectively. τ_{solvent} represents

Table 1
InP SQDs lifetimes with methanol (MeOH), chloroform (CF), acetone (Ac) and water (H₂O) solvents.

Environment	τ_1 (ns)	τ_2 (ns)	τ_3 (ns)
Pure N ₂	0.53	1.83	7.41
H ₂ O	0.56	1.91	7.54
MeOH	0.61	2.03	7.40
Ac	0.56	1.94	7.65
CF	0.56	1.93	7.44

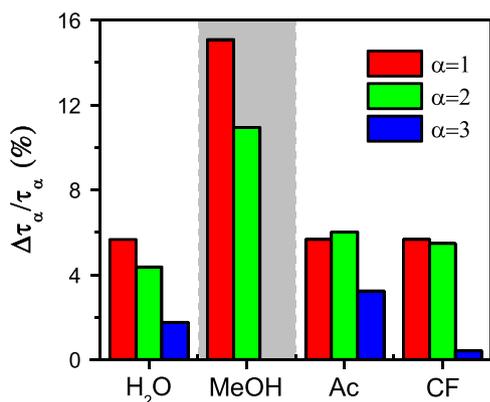


Fig. 5. Lifetime relative change $\Delta\tau_\alpha/\tau_\alpha\%$ of a series of common solvents. Namely, water, methanol, acetone and chloroform respectively. An increase in the decay constants (mainly τ_1 and τ_2) was observed when solvent vapours were present in the gaseous atmosphere surrounding the InP/InGaP SQDs. The amplitude of the effect is larger in the case of methanol (grey shaded area) respect to the other solvents.

the decay time when the sample is exposed to solvent vapour and τ_{N_2} is the decay time in the initial condition (pure N₂). The lifetime relative changes are reported in Fig. 5.

It can be noticed that methanol evokes a large stretching of the decay times when compared to other solvents at a fixed concentration of 2×10^4 ppm (with values of 15% and 11% for τ_1 and τ_2 , respectively). Conversely, all the other solvents cause a similar effect of a slight increase of both τ_1 and τ_2 , of approximately the same magnitude (circa 5–6%), almost irrespective of solvent type.

Interestingly, the long final decay time τ_3 is less influenced by the surface conditions produced by the different vapour solvents. This experimental evidence strongly supports the ascription of such component to the lateral coupling between the dots instead of the coupling between QDs confined states and surface states.

We checked also the reversibility of the sensing process after solvent exposure. The initial decay time is recovered in about 5 min of chamber purging with N₂ flux, that is the same recovery time we found in the case of the CW-PL sensing experiments [8].

The time resolved photoluminescence results strongly support the sensing mechanism model we previously hypothesized [8]. The target molecules present in the gas environment, passivating the non-radiative decay channels related to the QDs surface states, produce at the same time an enhancement of the PL signal in the steady state and an increase in the luminescence decay time. In our case the surface states are mostly represented by the dangling bonds that could be both positive In⁺ and negative P⁻ ions.

The effect of the different gas molecules on the optical properties suggests an interpretation about the nature of the interaction with the surface defects. As we discuss shortly below we believe that the involved defect type should be in prevalence negatively charged (donor), coming from the P atoms. Indeed, the molecules employed for the experiments present differences regarding the polarity, charge distribution and steric hindrance. In

fact chloroform is a solvent with a weak polarity, therefore its interaction with defects may occur by weak London dispersion force and therefore its effectiveness in the passivation of the radiative pathway is limited. Acetone is a polar aprotic species having a positive charge distribution rather spread on the carbon moiety with respect to the negative (around oxygen) one so it preferentially interacts with positive defects. As a consequence its limited effect on our system suggests that the involved defects are not positively charged. Moreover, its low response can be ascribed both to its low polarity and its large steric hindrance. Methanol produces the most noticeable effect and it is a polar and protic solvent. In this case the positive charge is rather localized on the hydrogen of the OH group which tends to interact with negatively charged species. On the other side, using water which is a very polar and protic molecule the effect we measured is very low. To explain such apparent inconsistency, a more complex representation of the observed phenomenon has to be taken into account where not only the interaction between solvent and surface defects is important but also the solvent–solvent intermolecular interactions. In fact water molecules tend to interact strongly with each other, forming a complex network of hydrogen bonds, methanol instead tend to be adsorbed on the surface as a monolayer. This could explain its higher effectiveness in luminescence enhancing modifying the optical properties.

4. Conclusion

Summarizing, we studied the chemical sensitivity of InP/InGaP SQDs by TR-PL spectroscopy. PL transients with and without vapours were fitted with a 3-exponential decay law (with average times of 0.5, 2.0, 7.0 ns). A very weak effect on the decay times for acetone, chloroform and water was found while a consistent effect was measured for methanol. In particular, the longer initial decay time (2.0 ns component) showed a relative increase as high as 15% in methanol when compared with the response in nitrogen environment. This result supports our previous model based on the ability of the organic molecules to saturate non-radiative QDs surface states. The mechanism we propose can satisfactorily account for both the observed experimental effects since by hindering the non-radiative channels a more intense CW-luminescence signal and a longer lifetime have been found. The presented results showed that InP/InGaP SQDs are a suitable material for application in optical chemical sensors, bio-sensors and lab-on-chip devices and indicate a possible way to increase their sensitivity.

Acknowledgements

The authors thank Prof. M. Paci (Chemistry Dept., University of Rome “Tor Vergata”) for the useful scientific discussions and F. Schnütte at the Leibniz-Institut für Kristallograpie Berlin for AFM measurements. The invaluable technical contribution of Dr. Michiel Hilbers of the Van’f Hoff Institute for Molecular Sciences is kindly acknowledged. The research leading to these results has received funding from LASERLAB-EUROPE (Grant agreement no. 284464, EC’s Seventh Framework Programme).

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