



# InP/ZnS quantum dots synthesis and photovoltaic application

Anna Pidluzhna<sup>1</sup> · Pavlo Stakhira<sup>2</sup> · Glib Baryshnikov<sup>3,4</sup> · Asghar Jamshidi Zavaraki<sup>5</sup> · Hans Ågren<sup>6</sup>

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## Abstract

In the present paper hybrid core–shell InP/ZnS quantum dots were prepared by the one pot synthesis method which does not require additional component injections and which complies more with cost requirements. The synthesized quantum dots were characterized by X-ray diffraction and optical spectroscopy methods. The applicability of the synthesized InP/ZnS core–shell particles in inverted solar cells fabricated with a step-by-step procedure which combines thermal vacuum deposition and spin-coating techniques was investigated. The resulting efficiency of the fabricated inverted solar cell is comparable to that of quantum-dot sensitized TiO<sub>2</sub> based solar cells. Therefore, hybrid core–shell InP/ZnS particles can be considered as multifunctional light-harvesting materials useful for implementation in different types of photovoltaic devices, such as quantum dot sensitized solar cells and inverted solar cells.

**Keywords** Quantum dots · Nanohybrid materials · Core/shell structures · Inverted solar cells

## Introduction

During the last decades, the synthesis and properties of colloidal quantum dots (QDs) have attracted a great attention provoked by the unique phenomenon of quantum confinement (Thambidurai et al. 2010; Bawendi et al. 1990; Kucur et al. 2003). Because of this effect, particles in the nanosize scale demonstrate properties very distinct from those in the bulk. As nanosize QDs exhibit a high surface to volume ratio, surface states play a critical role in their physical

and chemical behaviour and can significantly influence the absorption and luminescent properties. A wide list of recent reports confirm the potential for applications of QDs in photovoltaics (Beard et al. 2014; Hillhouse and Beard 2009), light emitting devices (Zhang et al. 2016; Ma et al. 2013; Kagan et al. 2016) and in many environmental (Tayebi et al. 2016; Abbasi et al. 2017) and biomedical experiments (Khan et al. 2017; Liu et al. 2018; Kulchat et al. 2018; Biju et al. 2010).

A significant progress in QDs synthesis and application in photovoltaics has been attained in the field of II–VI semiconductors (Cd-containing in particular) (Su et al. 2016; Rogach et al. 2008; Zeng et al. 2008; Barak et al. 2018), especially recently because of energy crisis and intensive search for novel materials for all branches of energy saving technologies (Jasiński et al. 2021; Grygorchak et al. 2020; Ivashchyshyn et al. 2021). However, the common inclusion of cadmium has rendered concerns about toxicity and cancer (Brunetti et al. 2013; McKittrick and Shea-Rohwer 2014). Therefore, the search for safer and less harmful materials but with the same favourable properties as cadmium QDs is a very urgent problem. The switch to III–VI semiconductors is a promising route to achieve this goal because of their narrow bandgap and large exciton Bohr radius and consequently they demonstrate stronger size effect (Brichkin 2015). Thus, InP materials are good candidates for development in photovoltaics, because they demonstrate suitable

✉ Anna Pidluzhna  
anna.pidluzhna@pcz.pl; ussurian@yahoo.com

<sup>1</sup> Faculty of Electrical Engineering, Czestochowa University of Technology, Al. Armii Krajowej 17, 42-200 Czestochowa, Poland

<sup>2</sup> Lviv Polytechnic National University, S.Bandera 12, Lviv 79013, Ukraine

<sup>3</sup> Laboratory of Organic Electronics, Department of Science and Technology, Linköping University, 60174 Norrköping, Sweden

<sup>4</sup> Department of Chemistry and Nanomaterials Science, Bohdan Khmelnytsky National University, Cherkasy 18031, Ukraine

<sup>5</sup> Faculty of New Sciences and Technologies, University of Tehran, North Kargar Street, Tehran, Iran

<sup>6</sup> Department of Physics and Astronomy, Uppsala University, 751 20 Uppsala, Sweden

optical properties combining size-tunable emission in the visible and near-infrared spectral ranges (bulk bandgap: 1.35 eV) and low intrinsic toxicity (Brunetti et al. 2013; Brichkin 2015; Tamang et al. 2016; Mushonga et al. 2012; Liu et al. 2008). While different synthesis procedures have been worked out for obtaining good quality InP nanocrystals (Mushonga et al. 2012; Liu et al. 2008; Allen et al. 2010; Gary and Cossairt 2013) there are still a few problems difficult to avoid. The strong bonding between In and P atoms makes the control on the formation and nanocrystal growth quite difficult. For this reason the synthesized nanocrystals have often shown poor monodispersity. The strong interaction also results in the formation of a large number of surface defects and, consequently, in poor optical properties and power conversion efficiencies (Yang et al. 2017; Cros-Gagneux et al. 2010; Wang et al. 2016).

One of the methods to avoid above mentioned problems is to synthesize protected InP/ZnS core shell QDs via a colloidal route, where a ZnS shell passivates the defect surface on an InP core. (Yang et al. 2017; Shang and Ning 2017; Zavaraki et al. 2020; Kumar et al. 2018; Li and Reiss 2008) Thus, the purpose of this work was to prepare the InP/ZnS QDs with the one pot synthesis method (Li and Reiss 2008; Park et al. 2007) which does not require additional component injections and among other features comply more with cost requirements. In this work we have also investigated for the first time the applicability of the synthesized InP/ZnS nanocrystals in inverted solar cells formed by a step-by-step vacuum deposition procedure in combination with a spin coating technique.

## Materials and methods

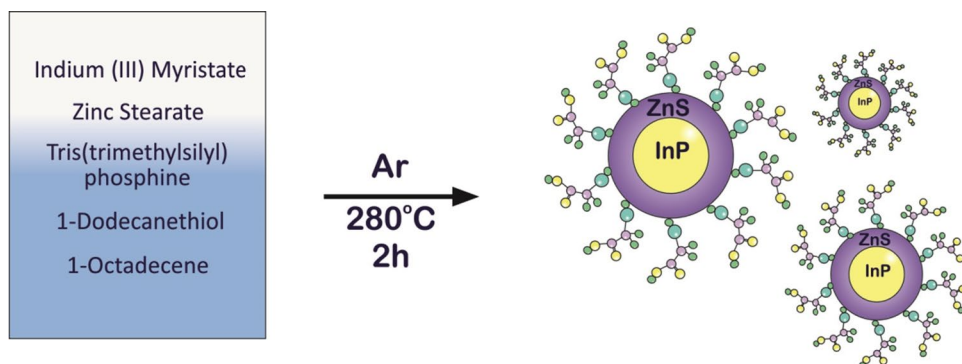
InP/ZnS core shell QDs were synthesised by a “heating-up method” using the single-step one pot synthesis procedure described by Zavaraki et al. (2020) with slight modification. The scheme of the reaction is presented in Fig. 1.

All reagents were of analytical purity grade. Indium myristate (0.1 mmol), prepared from indium acetate

and myristic acid (MA), tris(trimethylsilyl)phosphine (P(TMS)<sub>3</sub>, 0.1 mmol), zinc stearate (0.1 mmol), dodecanethiol (DDT, 0.1 mmol) and 1-octadecene (ODE, 8 mL), was mixed in a three-neck flask equipped with a vacuum pump. The oxygen dissolved in the reaction medium was removed from the system by an argon flow at room temperature, then the mixture was heated up to 280–290 °C and kept at this temperature for 2 h to let the InP formation with change of reaction medium colour into green and to grow the ZnS shell. Then, the mixture was cooled down to room temperature and 1 volume equivalent of a chloroform/methanol (1:1 vol:vol) mixture and 10 equivalents of acetone were added to stabilize the QDs. The washing procedure followed by centrifugation at 12,400 rpm was repeated 3 times and the final nanocrystals were dispersed in toluene for further use. The optical absorption of the QDs was measured by a UV–visible spectrophotometer and their crystallinity was evaluated by X-Ray Diffraction. Fluorescence lifetimes were recorded using the time-resolved single photon counting (SPC) method using an EPLED 340 nm picosecond pulsed light emitting diode as an excitation source.

Regioregular poly(3-hexylthiophene) labeled as P3HT M101 with the following characteristics; Mw = 65,500 and Mn = 32,000 and (6,6)-Phenyl-C<sub>61</sub>-butyric acid methyl ester (PCBM), was acquired from Ossila, glass/ITO substrates and the Al wires were purchased from Sigma-Aldrich. QD-based photovoltaic devices were prepared with step by step procedure which combines the thermal vacuum deposition method and spin-coating technique. The glass/ITO substrates were prepared by washing with distilled water, 2-propanol, NaOH, DI water in series and dried with hot dry air flow. The overall scheme of the inverted structure for the fabricated solar cells is ITO/PCBM(57 nm)/QD(5 nm)/P<sub>3</sub>HT(50 nm)/MoO<sub>3</sub>(2 nm)/Al(100 nm). The thickness of the thin films was controlled and determined by means of a profilometer (Dektak XT, Bruker). The current density–voltage dependences and the photovoltaic characteristics of the fabricated cells were examined using the HP4145A semiconductor parameter analyser and the ABET technologies

**Fig. 1** Scheme of InP/ZnS QDs synthesis by “heating-up method”



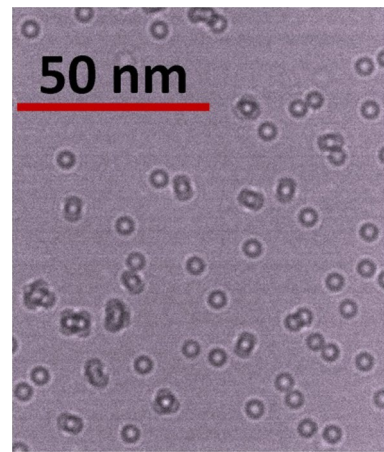
Solar simulator (for the standard AM 1.5G solar spectrum, the total available power is  $982 \text{ W/m}^2$ ).

## Results and discussion

After ZnS shell coated InP quantum dots were synthesised a structural characterisation of the nanocrystals was carried out by the X-ray diffraction (XRD) technique. The XRD spectrum is shown in Fig. 2, displaying peaks corresponding to (111), (200) and (311) lattice planes which after comparing with zinc blend structures of InP (JCPDS # 32-0452) and ZnS (JCPDS # 80-0020) can be attributed to the cubic InP phase (Mushonga et al. 2012; Cros-Gagneux et al. 2010).

The peaks of the diffraction patterns are clearly distinguishable, indicating a crystalline nature of the QDs, but still a bit broad due to the nanosize of the synthesised material. Nevertheless, the average particle size was evaluated by application of the Scherrer equation for (111) maximum and it is about 3 nm. While no individual peaks for ZnS were found, probably because the ZnS shell is too thin, no other new peaks were observed and hence we assume that the structure is retained as InP/ZnS. The results of the XRD characterization agree well with the previously reported data for analogous InP/ZnS QDs. (Cros-Gagneux et al. 2010; Zavaraki et al. 2020; Kumar et al. 2018; Li and Reiss 2008; Ryu et al. 2009; Angel-Huerta et al. 2018; Samal et al. 2013; Virieux et al. 2012; Kim et al. 2012).

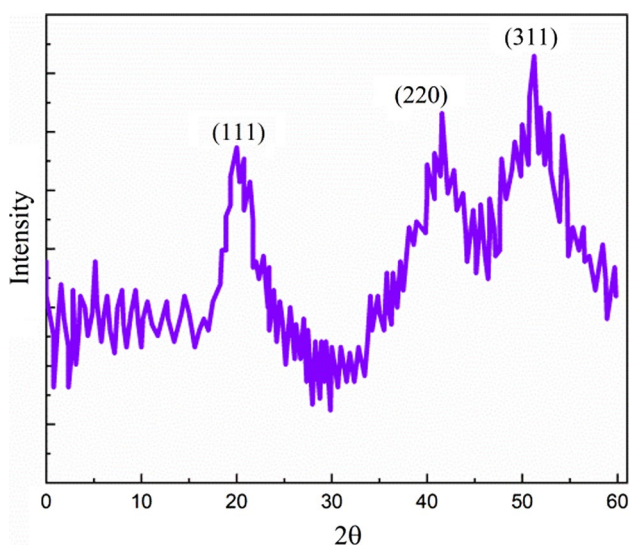
In addition to the X-ray diffraction analysis the transmission electron microscopy (TEM) was used to check the quality and estimate the size of the synthesised QDs and Fig. 3 shows that the shape of InP/ZnS QDs is spherical and the



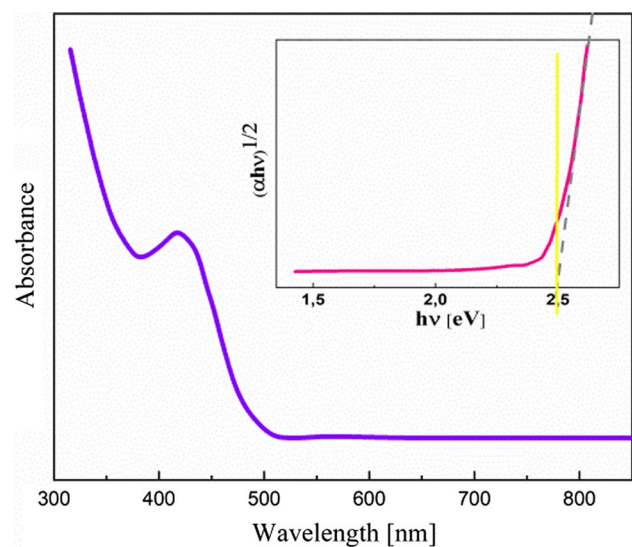
**Fig. 3** TEM image of synthesised InP/ZnS QDs

size is around 4–6 nm which is very close to the average particle size estimated from the XRD pattern.

The optical properties of the synthesised QDs were analysed with a UV–visible spectrophotometer. The effects of the synthesis procedure on the optical properties of the InP/ZnS nanocrystals are presented in Fig. 4. Analysing the absorption spectra one can confirm the formation of core/shell type InP/ZnS QDs (Su et al. 2016; Wu et al. 2013; Biadala et al. 2016; Yang et al. 2018). It is well-known fact that semiconductor materials in the nanometric scale absorb light strongly when the excitation energy is greater than the bandgap energy. During this process electrons get enough energy to be transferred from the valence band to the conduction band. The energy which is needed for the electron transfer depends on the size, structure type and nature of the QD and can be quantified with measurements



**Fig. 2** Peaks in the XRD pattern of InP/ZnS core/shell sample



**Fig. 4** Absorption spectrum and Tauc plot for ZnS-coated InP QD

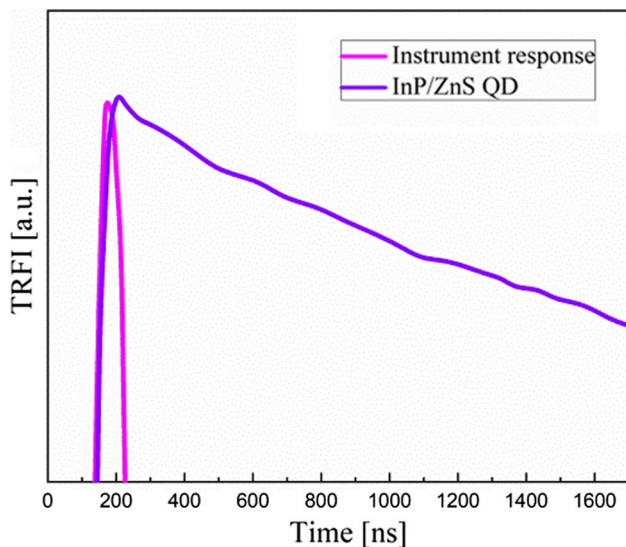
of UV–Vis spectroscopy. The first observable peak in the absorption spectrum depicts the lowest energy state of the excited electrons.

The QDs show an absorbance maximum at 420 nm and the bandgap energy (Fig. 3) for the synthesised InP/ZnS QDs is 2.5 eV which agrees well with other works (Mainet et al. 2012; Reiss et al. 2009; Davar et al. 2015). The absorbance band ~400–430 nm is rather narrow which confirms quite small size distribution of synthesised QDs (Ryu et al. 2009; Ziegler et al. 2008).

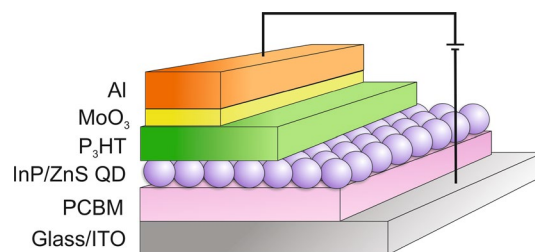
To study the excited state dynamics the QDs were analysed by fluorescence spectrophotometry. The fluorescence emission decay of the synthesised ZnS-coated InP QDs is illustrated in Fig. 5. It lies in the microsecond region showing fluorescent behaviour which is attributed to type I band alignment in the InP–ZnS heterostructure, where the narrow bandgap InP core is surrounded by the wide bandgap ZnS shell and to that both electron and hole are confined in the conduction and valence band of the core, respectively (Zavaraki et al. 2020).

To investigate the photoactive properties of the synthesised QDs and their applicability in solar cells the well-established P3HT/PCBM system was chosen because of its good hole and electron transport properties of the P3HT and PCBM materials, respectively (Garcia-Belmonte et al. 2010; Bisquert et al. 2008).

The inverted solar cells (ISCs) of the ITO/PCBM/QD/P3HT/MoO<sub>3</sub>/Al architecture (Fig. 6) with synthesised QDs were formed with a step-by-step procedure. The electron transport PCBM layer was deposited by the thermal vacuum deposition technique onto glass/ITO templates, then the solution of InP/ZnS QDs in toluene was spin-coated on the



**Fig. 5** Time-resolved fluorescence intensity decay curve for ZnS-coated InP QDs



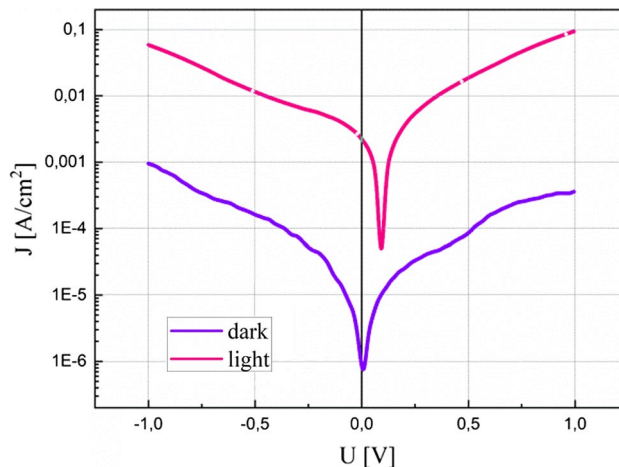
**Fig. 6** Schematic Solar Cell architecture

surface of PCBM and then MoO<sub>3</sub> and P3HT hole transport layers and Al electrode contacts were thermally deposited in vacuum again.

The inverted, not direct, structure of the solar cell was chosen, because the optically active behaviour of the synthesised quantum dots [not the behaviour of P3HT which absorbs light in the ~400–600 nm range (Bisquert et al. 2008)] was of scientific interest. In the as fabricated OPV device the light illumination was applied from the side of the ITO and quantum dots and not from the side of the P3HT and metal contact; therefore, the inverted structure was chosen and executed in such a configuration (Fig. 5).

The current–voltage (J–V) characteristic of the fabricated device is shown in Fig. 7—the device parameters are summarized in Table 1, where  $V_{oc}$  is the open circuit voltage,  $I_{sc}$  is the short circuit current, FF is the fill factor and  $\eta$  is the power conversion efficiency.

It is noteworthy that the power conversion efficiency of the investigated ISC is comparable with the power conversion efficiency of quantum dot sensitized solar cells on the basis of InP/ZnS quantum dots which were described in Zavaraki et al. (2020). This fact indicates the universality of InP/ZnS quantum dots and their applicability as optically



**Fig. 7** J–V characteristics of device sensitized with ZnS-coated InP QDs (pink curve) upon light irradiation and without it (blue curve)

**Table 1** Average values of photovoltaic parameters and standard deviations estimated for the ZnS-coated InP-based solar cell device

Parameter	value
$V_{oc}$ , mV	$95 \pm 8$
$J_{sc}$ , mA	$0.550 \pm 0.001$
$V_{max}$ , mV	$75 \pm 4$
$I_{max}$ , mA	$0.503 \pm 0.001$
FF	$0.260 \pm 0.004$
$\eta$ (%)	$0.36 \pm 0.03$

active components in solar cells of different configurations. Hence, until today the values for power conversion efficiencies for QD-based solar cells are not so high, we believe that the further progress in InP/ZnS QDs application in ISCs can give important perspectives concerning the problems of controllable size distribution synthesis, core passivation and shell formation.

## Conclusions

Cd-free colloidal InP/ZnS core shell QDs were successfully synthesised in this work by the “heating-up” one pot method. The results of UV–Vis absorption spectroscopy confirm that synthesised QDs can be attractive for application in the field of photovoltaics. For the first time to our knowledge, an inverted solar cell structure ITO/PCBM/QD/P3HT/MoO<sub>3</sub>/Al has been formed with a step-by-step procedure and its photovoltaic behaviour demonstrated. The value for the power conversion efficiency of the presented ISC is comparable with that for other InP/ZnS QD sensitized solar cells and indicates the universality of synthesised InP/ZnS quantum dots for application in devices of different architectures.

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**Authors' contributions** AP: methodology, investigation, data curation, visualization, writing—original draft, reviewing and editing. PS: conceptualization, supervision. GB: validation, writing—reviewing and editing, project administration. AJZ: synthesis, investigation, formal analysis. HÅ: project administration, writing—review and editing, supervision, funding acquisition.

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**Availability of data and materials** Not applicable.

## Declarations

**Conflict of interest** The authors have no competing interests to declare that are relevant to the content of this article.

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