Direct Optical Patterning of Semiconductor Quantum Dots

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Contributor: Francesco Antolini

Patterning, stability, and dispersion of the semiconductor quantum dots (scQDs) are three issues strictly interconnected for successful device manufacturing. Several authors adopted direct optical patterning (DOP) as a step forward in photolithography to position the scQDs in a selected area. However, the chemistry behind the stability, dispersion, and patterning has to be carefully integrated to obtain a functional commercial device.

semiconductor quantum dots ligands

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1. Introduction

Semiconductor quantum dots (scQDs) closely related to their function, which is in turn linked to their stability and homogeneity. For real exploitation of the scQDs in a commercial device, this material should have three main characteristics, i.e., (i) stability in the conditions of usage, (ii) homogeneity of distribution within the device to guarantee uniform performances, and, for some applications, like in displays, (iii) the possibility to be patterned. These three characteristics, however, cannot be taken alone, because they are interconnected. Indeed, the development of the chemical processes that underlies the stability and dispersion has to be compatible with the adopted patterning technology.

The patterning strategies of scQDs can be divided into three main groups: photolithography [1], contact printing[2], and ink-jet printing [3]. Some recent developments in patterning are exploring the direct use of light as a tool for patterning as in the “classical” photolithography, but to control the position of the object of interest, the scQDs, without the use of masks and repeated steps of layer curing/etching typical of the photolithographic process. If, on one side, the direct optical patterning (DOP) of scQDs simplifies the process of patterning, on the other side this shifts on the material side the critical issues of the scQDs’ dispersion, protection, and patterning itself. Indeed, the role of light is to block the scQDs in a specific position by creating a network that sticks them, by changing the scQDs solubility or by growing them directly.

1.1 The Semiconductor Quantum Dots

Semiconductor quantum dots [4] (scQDs) are among the most studied and utilized nanomaterials because their compositional and morphological tunability modulates their optoelectronic properties that can be adapted for different applications. On the other side, the relatively easy synthesis in a colloidal state further contributes to their real-world use.

There are two main approaches for semiconductor QD fabrication [5]: the physical methods and the wet-chemical methods.

The physical methods include the molecular beam epitaxy (MBE) and the metal-organic chemical vapor deposition (MOCVD) that allow the preparation of thin layers of semiconductor QDs or deposit them over a wafer.

The chemical methods involve the synthesis in the liquid phase (organic solvent or water) at relatively high temperatures (100–350 °C) in the presence of precursors and surfactants. The controlled combination of the precursors, their ratio, the presence of surface ligands, the reaction temperature, and the duration of the heat treatment determines the stoichiometry, size, and shape of the scQDs. The atomic species forming the scQDs belong to the elements from II–VI, III–V, IV–VI, and IV groups, and include the metal halide perovskites ($CsPbX_3$ X = I, Br or Cl) [6][7][8].

The possibility to control the electro-optical properties of the scQDs through all of these parameters without changing the whole chemistry of the system is a key advantage of this class of materials and opens many research paths [9]. However, finding the right combination of them is not an easy task, especially in the case of core/shell systems. For example, a careful selection of precursors and ligands is strictly connected with the temperature of nucleation and growth of the scQDs, and together their optical properties. For example, if the growth and nucleation processes are not well distinguished final scQDs are not monodispersed (monodispersion means that all the scQDs have the same size) and, hence, the FWHM of the preparation is broad. In the same way, the high crystalline quality of the core ensures a high quality of performance [10].

The modulation of the density of states, i.e., the tuning of the scQDs' electro-optical properties, is another technological aspect that is deeply studied. One strategy is to modify the shape of the nanocrystals, while another research direction is the doping of scQDs as reported by Mocatta et al. [11].

Another critical aspect of the scQDs' synthesis that influences their application in thin films is the role of the ligands that mediate the interaction with the substrate and with themselves.

1.2. The Quantum Size Effect and Its Role in the Modulation of the Electro-Optical Properties of the scQDs

The importance of this class of nanomaterials lies in the possibility of modulating their optoelectronic properties by their size, composition, and architecture. The modulation of the optical properties by size is the so-called quantum size effect [9]. This effect takes place when the size of the semiconductor becomes smaller than the wavefunction of the exciton (the electron and hole pair formed by Coulomb interaction) [12], typically below 10 nm, depending upon the type of material [9]. In this condition, the bandgap of the semiconductor material becomes quantized and the effective bandgap increases, decreasing the particle size and modifying the absorption and emission properties (**Figure 1**).

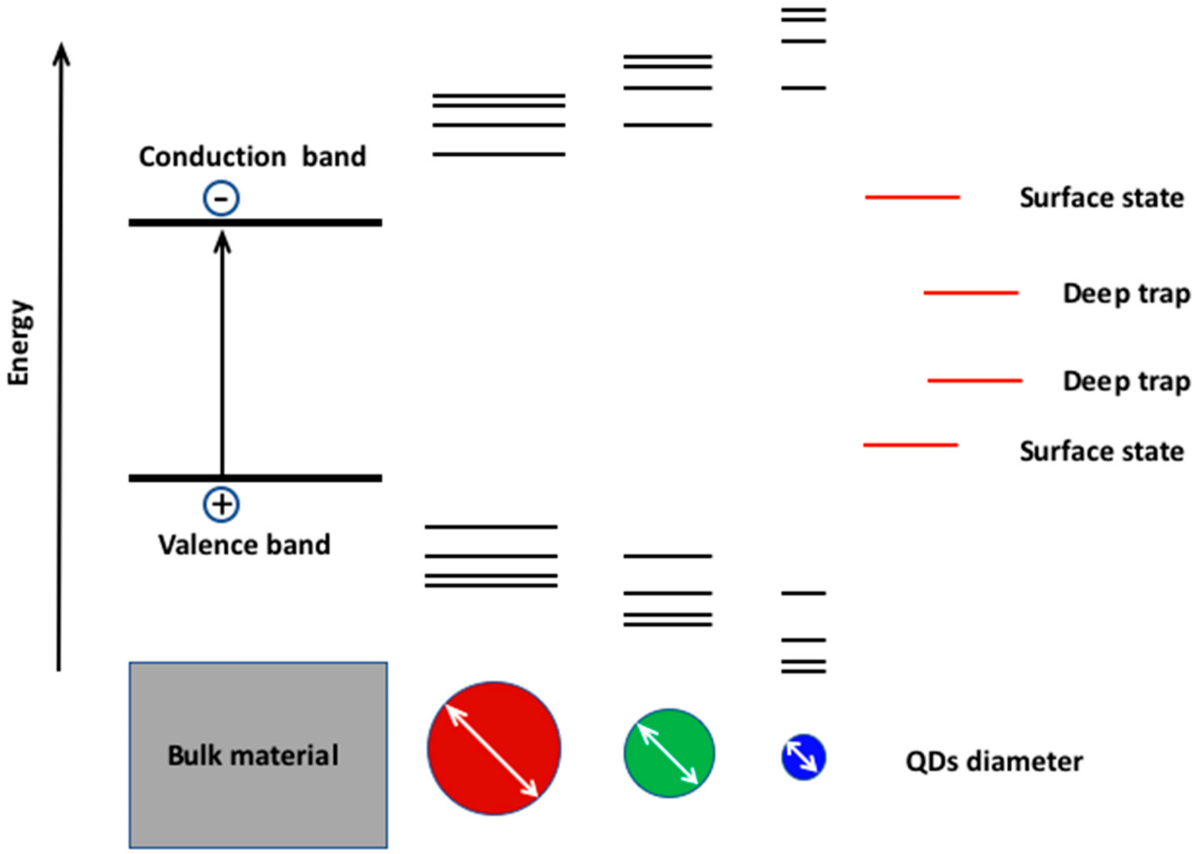

Figure 1. The valence band and the conduction band of the semiconductor bulk material become quantized (black bars) when the size of the QD (white arrow) becomes smaller than the Bohr diameter. The defect surface states and deep traps (red bars) are due to surface defects and crystal defects.

In such a way, the bandgap of the scQDs can be tuned from different energy levels from the ultraviolet to infrared range. The bandgap fine-tuning enables the specific emission wavelength, the size uniformity of the emitting cores causes the narrow emission, while the absence of crystal defects mainly at the surface (**Figure 1**) ensures a high photoluminescent quantum yield (number of emitted photons per absorbed photons) and also contributes to narrow emission.

The wavelength tunability (color selection), the narrow emission (color purity), and the photoluminescent quantum yield (brightness) are characteristics of paramount importance for the application on a device. The improvement of the knowledge of the scQDs showed that the source of the problems are the defects states from surfaces of the scQDs (surface traps and deep traps) that perturb the band gap structure, changing the emission wavelength and lowering the luminescent quantum yield.

1.3. The Core@shell Systems

The solution for the stabilization of the scQDs structure arrived from the introduction of the core@shell systems [13][14][15]. In this configuration, the surface defects are fixed by growing an inorganic shell over the scQD core. The shell role is two-fold: the passivation of the surface defects and the localization of the exciton into the core [5]. The growth of another type of material over the scQD should be chosen carefully because the shell material should crystallize over the core without introducing any mechanical (crystallographic) stress, which means more surface defects.

Another crucial point for the realization of the desired core/shell system is the reaction methodology. It is possible to summarize three different methods of growth of the core/shell systems; namely, the two (multiple)-steps synthesis, the one-pot synthesis, and the SILAR (successive ion layer adsorption and reaction). The two-steps reaction is the most used approach because it allows the removal of the reaction byproducts after each reaction. Cao et al. [16], for example, prepared a multiple core/shell/shell system like CdSe/CdS/ZnS with this approach. First, they synthesize the core that is used, after the purification, as a reagent for the synthesis of the shell. The CdSe/CdS is then purified and utilized for the growth of the ZnS final layer. In this case, the metal precursors are CdO, $Cd(OAc)_2$, and $Zn(OAc)_2$, while the selenium and sulfur are added slowly to the reaction mixture. The slow addition of the chalcogenides prevents the nucleation of the shell material. The SILAR methodology forecasts the formation of the shell layer-by-layer. Each layer is realized by two different injections in the reaction vessel of the cationic and anionic precursors [17].

The one-pot synthesis approach consists of the formation of the scQDs using a core with a gradient shell. W.K. Bae et al. [18] introduced this approach, suggesting that the growth of this type of material, indicated as $Cd_{1-x}Zn_xSe_{1-y}S_y$, is due to different reactivity of the reagents (metal and chalcogenides) mixed in the same vessel when reacting at the same temperature (the reaction is carried out at 300 °C). All of these reaction approaches are widely used by researchers that developed many particular variations, always to improve the photoluminescent quantum yield (PLQY), the FWHM, and the scQDs’ stability.

Cao et al. [16] sorted out a CdSe/CdS/ZnS scQD with an electroluminescence efficiency six orders of magnitude higher than the standard ruthenium complex. That can be used for an electrochemical immunoassay for the development of QD-LEDs. Hanifi et al. [19] set up a method of core/shell synthesis that produces CdSe/CdS scQDs having a PLQY approaching 1. These scQDs with this high PLQY are developed for applications in the photovoltaic field.

Other examples of core-shell systems are the so-called giant QDs. They are systems with a very high shell/core volume ratio (shell thickness higher than 1.5 nm) and the absorption is dominated by the shell [20][21]. These systems are particularly advantageous because they exhibit a high distance between the absorption and emission maxima (Stokes-shift) that improves the efficiency of the optoelectronic devices where these systems are included.

A further example of this band engineering for the optimization of optoelectronic properties is the modulation of the shell shape, like the dot in rods [22][23] and nanoplatelets [24]. Both engineered structures show a high Stokes-shift between the absorption band and emission band that ensures the absence of the reabsorption of the emitted light [25]. Both types of structures were described in this section because the ligands play a pivotal role in their preparation.

2. scQD Dispersion: The Ligands and the Surrounding Environment of the scQDs

The ligands mediate the compatibility of the scQDs within the matrix. The best way to enhance the dispersion is to prepare an scQD with a ligand compatible with the host matrix [26]. The dispersion of the scQDs in a matrix is a particularly important factor for the scQDs' application, especially in a solid state like in a film, because the aggregation phenomena quench the electro-optical properties of the scQDs[27], nullifying all of the efforts made to obtain nanocrystals with excellent optical properties.

3. QD Stability: The Effect of Oxygen and Moisture

Answering the question about the stability of the scQDs under ambient conditions in combination with light will help to adopt the necessary countermeasures to improve the life of any device equipped with this material.

Only recently, the group led by Peng clarified the role of oxygen[29] and water[30] by studying systematically their effect on a well-defined system, the CdSe/CdS core/shell scQDs, in defined experimental conditions in terms of atmosphere (only oxygen, only water, or their defined combination) and different phases at single scQD level or as an ensemble of scQDs in thin film and solution.

4. Stabilization of the QDs at Collective Level

The scQDs' deterioration, i.e., the loss of the ligands and atoms from their structure, can be prevented by adopting two different strategies: (i) the encapsulation of each single scQD and (ii) the encapsulation of the scQDs embedding them within a matrix.
In the former, the main idea is to overcoat the single QD with a material that "freezes" its structure and hence its properties so that they can be incorporated into the final device. In the second approach, they are dispersed in a covalent network that does not protect the
single scQDs, but all of the scQDs.

4.1. The Thiol-Ene Network

A common way of collective encapsulation of the scQDs within a matrix is to use molecules forming a network through the thiol-ene chemistry [33][34].

4.2. The Polymers

The collective encapsulation with polymers was widely used [36]; however, the direct mixing of polymers and scQDs, even if it is relatively simple, suffers from the issue of scQD aggregation. When there is a chemical interaction between the scQDs and the polymer, the encapsulation strategy is more effective. Lesnyak's group gave an interesting example of how these two obstacles, dispersion and stability, can be circumvented by using polymers [37].

4.3. The Siloxanes

The siloxane organic–inorganic hybrid materials [38] (SHMs) are compounds that have the tetravalent silicon bonded with oxygen and one or more bonds replaced by a covalent linkage with an organic substituent [39]. With this kind of chemistry, the siloxane polymers bear ceramic-like properties joined with the ones of the organic materials. From the functional point of view, this fact means that these materials display both the ceramic “character”, i.e., high-temperature stability, hardness, chemical resistance, and optical transparency, combined with the organic “character”, i.e., low temperature and solution processing, modulation of matrix porosity, and flexibility. Here will be described the capacity of siloxanes to act as a collective encapsulating agent with high chemical thermal and mechanical stability, as recently presented by Bae’s group for the stabilization of scQDs in different conditions [40][41].

5. Quantum Dots Direct Optical Patterning (DOP)

The research on the patterning technologies of scQDs is an active area of study for their industrial application due to the high interest of companies, especially in display manufacturing [36]. Photolithography is the most widely used technique in the industrial field; however, the use of photoresists and the multiple steps of etching/washing can alter dramatically the QDs’ functionality and the production costs, respectively. Recently, different authors published some works that utilize direct optical patterning (DOP) as a step forward in photolithography.

5.1. Direct Optical Patterning of scQDs with Thiol-Ene Cross-Linkers

In Section 4 the effect of the thiolene encapsulation on scQDs stability was already reported. However, the same chemical process can be adopted for the photolithographic patterning of perovskite scQDs as shown by Zhang et al. [43].

5.2. Direct Optical Patterning of scQDs with Siloxanes

The possibility using the DOP combined with the siloxane chemistry to pattern and protect the scQDs has been readily demonstrated by Bae’s group for the manufacturing of quantum dots color filters (QD-CF) for displays [44].

5.3. Direct Optical Patterning via In Situ Ligand Exchange (DOLFIN)

The patterning strategy that uses the ligands of the scQDs as active molecules for the patterning itself was proposed by Talapin’s group [45][46] (direct optical lithography of functional inorganic nanomaterials DOLFIN).

5.4. Direct Photolithography of scQDs via Photo-Active Cross- Linkers

Another DOP strategy in which the photoresist/matrix is absent is the one using the azide cross-linkers [47][48]. The approach is similar to the one explored with the polymers and siloxanes but uses only a cross-linker molecule while the matrix is absent. The role of the cross-linker activated by light is to create a network between the scQDs through their organic ligands.

5.5 Direct Photolithography of scQDs via Their Direct Synthesis

The direct synthesis of the scQDs using a laser within a film [51] can be considered belonging to the DOP strategies. The main technical difference with respect to the previous methodologies is that the laser was used to directly synthesize the scQDs. This technique, often called direct laser patterning, combines the flexibility of the laser technique both at the industrial and technological level with the chemistry and optical properties of the QDs.

6. Conclusions

Direct optical patterning (DOP) is an emerging tool to simplify the patterning process of the scQDs for display manufacturing that, however, is strictly related to the chemical processes that lead to the stability and homogeneity (dispersion) of the QDs for the correct function of the device. The optimization of the dispersion of the scQDs within a matrix is the golden rule to homogenize the interaction of the scQDs with the matrix itself. This means that the organic ligand at the QD surface should have the same chemical nature as the matrix. On the other side, stability is ensured by the formation of a close network of covalent bonds that cages the scQDs, preventing the loss of the ligands and surface atoms and, hence, preserving their optical properties. Considering these two main boundary conditions, five different approaches of DOP that exploit different chemical processes are evaluated.

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