Synthesis and perspectives
of complex crystalline nano-structures

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Research on inorganic colloidal nanocrystals has moved from the synthesis of simple structures, such as spherical nanoparticles, to more elaborate particles with shapes such as rods, stars, discs, and branched nanocrystals, and recently to nanoparticles that are composed out of sections of different materials. Nanocrystal heterostructures represent a convenient approach to the development of nanoscale building blocks, as they merge sections with different functionality in the same particle, without the need of inorganic cross-linkers. The present article gives an overview of synthesis strategies to complex nanocrystals and will highlight their structural properties, as well as discuss some envisaged applications.

1   Introduction

Synthesis techniques for colloidal nanocrystals have seen a fast development over the last years. In the nanometer size-regime the particles of some materials experience already quantum confinement effects. Therefore, by simply varying the size of a particle one can tune its properties over a considerable range, especially in the case of fluorescent semiconductor nanoparticles [1]. The relative ease of fabrication and manipulation combined with the flexibility offered by the possible tuning of their physical properties makes nanomaterials an appealing tool for many applications. This ranges from the development of LEDs [2] solar cells [3–5], lasers [6, 7], to biological tagging with semiconductor particles [8–10] and to biomedical applications of magnetic nanoparticle [11, 12] and also to future electronic devices [13–17].

A recent direction in the field of nanocrystal research is the development of synthetic techniques that allow for controlling the shape of the nanoparticles [18, 19]. Shape-controlled nanoparticles are promising candidates for several new applications. It has been proven for example that semiconductor nanorods enhance the efficiency of solar cells compared to spherical nanocrystals [5]. There are also several fundamental studies on the electronic behavior of semiconductor nanorods [20] and tetrapods [21] as these posses more intriguing shapes with respect to spherical nanocrystals, which might prove advantageous in displays, solar cells, field emitters and in nano-transistors. In addition, by introducing nanoparticles with a complex three-dimensional shape into composite materials, one can expect a strong influence on the materials' mechanical behavior.

The next step in complexity of nanoparticles is the fabrication of hybrid structures. The motivation for research in this direction is clear, as by merging different materials into a single nano-object, such object

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might exhibit multiple functionalities. First results have been demonstrated in this field, namely in the fabrication of hybrid materials. In this review we will focus on inorganic colloidal nanocrystals synthesized in hot organic solvents (200–400 °C).

2   Synthesis of spherical nanocrystals

Generally, the synthesis at high temperatures offers the advantage that defects are easily annealed from the crystal lattice. In addition, the nucleation and growth stages can be controlled and tuned easily. The reaction of the particles is started by injecting molecular precursors (which contain the atoms out of which the particle will be made) into a mixture of different organic molecules at a given temperature. These molecules have multiple roles. First, they serve as a solvent for the growing particles. Therefore, they have to support the high temperature without degradation and with negligible evaporation. Second, they also act as surfactants. At the high temperatures of the growth, they bind dynamically to the surface of the particles and by this they control the growth kinetics. When the synthesis is stopped by lowering the reaction temperature, such molecules form a stable monolayer around the particles. For a general overview on synthesis techniques we refer to various reviews [22, 23]. In a very simplified model, the surfactant molecules bind to and unbind from the surface of the nanoparticles, whereby the rates of these two processes are controlled by the temperature. Whenever there is a free site on the surface of a nanocrystal, the surfactants have to compete with the ions/atoms coming from the bulk of the solution for attachment to this site. In order to have a sufficient control on the dynamics of the growth, the surfactants must not bind too strongly to the particle surface, as this would suppress the crystals growth. On the other hand they should not bind too weakly, as otherwise the growth would be uncontrollably fast. This ultimately would result in the formation of large clusters and aggregates of clusters. The surfactant molecules most frequently used for such purpose are for instance phosphine oxides (tri-n-octylphosphine oxide) [1, 24], phosphines (e.g. tri-n-butylphosphine)) [1, 24], phosphonic acids [25], amines [24, 26], and carboxylic acids [27]. In general, nanocrystals of different materials require different surfactants to be grown in a controlled and reproducible way. CdSe, for instance, is frequently grown in a mixture of tri-n-octylphosphine oxide and tri-n-octylphosphine [1], whereas $CoPt_3$ is grown in a mixture of amines, carboxylic acid and hexadecandiol [26], and Fe_2O_3 in oleic acid [27]. At the end of the growth process, when the temperature of the reactor is lowered to room temperature, unreacted excess precursors can be removed by subsequent precipitation and re-dissolution of the particles, by adding solvents to which the surfactant-coated nanocrystals are not soluble. The resulting nanoparticles are hydrophobic because of the monolayer of surfactants bound to their surface. These particles are soluble in many organic solvents and can be also stored as a powder and re-dissolved at whish.

Spherical nanoparticles can be grown for a large variety of different materials. Due to their electronic structure semiconductor materials (such as CdSe, CdS, ZnSe, InAs, CdTe, etc.) are of special interest [28]. These nanoparticles show a size-dependent narrow fluorescence peak. In the case of CdSe, for instance, such peak can be centered anywhere within the visible range from 450 nm to 650 nm, depending on the particle size. Semiconductor nanocrystals also exhibit a size-dependent, continuous absorption spectrum, which is blue-shifted with respect to their bulk counterpart. This clearly distinguishes them from organic dyes, which possess a narrow absorption band at energies slightly higher with respect to their emitted energies. These optical properties and also their superior lifetime [29] compared to fluorescent dyes make semiconductor nanoparticles an attractive tool in fluorescent labeling techniques [30] [31, 32], as they render it easier to observe simultaneously different colours with only one excitation wavelength [8, 10]. Recently, even smaller particles of CdSe have been produced [33, 34] that emit in the blue range. Besides fluorescent, also magnetic particles as $CoPt_3$ [26, 35] or Fe_2O_3 [27] are interesting for many technical applications, such as drug targeting [36, 37] and magnetic storage devices [17].

A more detailed understanding of the growth mechanism of nanocrystals can be obtained in the general framework of the diffusion-controlled growth model [38, 39]. Nanocrystals are crystals and therefore possess facets. One can show that the dynamics of the growth depends on the surface energy of the different crystalline facets onto which new atoms are deposited [40, 41]. In the simplest case, we can

assume that the surface energy for all facets is nearly the same, and in most cases this is a reasonable assumption, as usually the nanocrystals grow isotropically (they have a rather spherical shape). In the theory of colloidal growth one can derive a key parameter, namely the "critical particle size", which is inversely proportional to the concentration of monomers in solution. A particle that has exactly such critical size is in equilibrium with the bulk solution of monomers, therefore monomers unbind and bind at the same rate on it, and thus this particle is not growing nor shrinking. Particles smaller than this critical size exhibit a negative growth rate (they tend to dissolve), whereas particles larger than the critical size are continuously growing. A general equation describing the distribution of growth rate versus size shows a maximum in growth rate for particles which are twice as big as the critical size. Therefore, during the synthesis one should try to arrange the distribution of sizes and the critical size in such a way that even the smallest particles present are significantly larger than the critical size. In this case the smaller particles grow faster than the bigger particles, resulting in a focusing of the size distribution over time [42].

One major complication is that, while the synthesis of nanocrystals is proceeding, the monomers in solution are consumed. This leads to an increase in the critical size and the reaction can ultimately shift into the so-called Ostwald-ripening [43] regime. Here the size distribution is such that the smallest particles present are smaller than the critical size. As soon as these small particles dissolve, they free monomers that can be incorporated into the bigger particles. This regime is characterized therefore by a fast broadening of the size distribution. For this reason, the critical size should be kept always small in order to obtain a narrow distribution of sizes in the final sample. One possibility is to stop the synthesis well before it enters into the broadening regime. Another possibility is to continuously add monomers to the solution [1, 42].

3 Shape control

The assumption of equal surface energies for all facets is most certainly an oversimplified model. For II−VI semiconductor nanoparticles in wurtzite structure, for instance, it has been proven experimentally [44] and theoretically [40, 41] that the different facets exhibit distinguishably different binding strengths for certain surfactant molecules. Especially phosphonic acids are strongly binding to the lateral facets of the wurtzite crystals CdSe, CdS and CdTe, whereas their binding efficiency to the basal facets, that is the (0001) and the (000$\bar{1}$) facets, is much lower. Therefore, the basal facets are more unstable than the prismatic facet and these two types of facets will grow at different rates, depending on the monomer concentration in solution. If the concentration of monomers in the growth environment is high, the basal facets will grow faster, and a rod shape will develop [45]. On the other hand, if the system is in the Oswald ripening regime (monomer concentration is low) and the nanocrystal is shrinking, the basal facets will dissolve at a higher rate than the lateral facets. The synthesis of the rod-shape is well controlled in the case of CdSe, CdS and CdTe. One additional point about the wurtzite structure is that there is also a remarkable difference between the (0001) and (000$\bar{1}$) facets. In the first case the cations expose three dangling bonds, in the second case only one (the opposite is true for the anions). Therefore the two facets are chemically different and have different growth rates [46].

A more complex shape is the tetrapod [19]. In this structure four rods are assembled into one three-dimensional object. A variety of materials may be grown in this shape, e.g. ZnO [47], ZnSe [48], CdS [49], CdSe [50] or CdTe [19, 51]. For type II−VI semiconductors there are two different crystallographic models that can rationalize this structure. In both cases the arms are described as rods that are growing in wurtzite structure. The two models differ in the way they describe the core out of which the arms are growing. The first model relies on the polymorphism of the material, most II−VI and some III−V semiconductors can grow both in the sphalerite and in wurtzite phase [52]. In this model the core consists of a sphalerite particle. The arms, which are in hexagonal phase, branch out of this core through a transition from the cubic to the hexagonal phase. The cubic core has four equivalent (111) facets that are chemically identical to the (000$\bar{1}$) facets of the hexagonal phase. Therefore, through generation of a stacking fault the growth can shift from the cubic to the hexagonal phase. Growth along the (0001) direction is
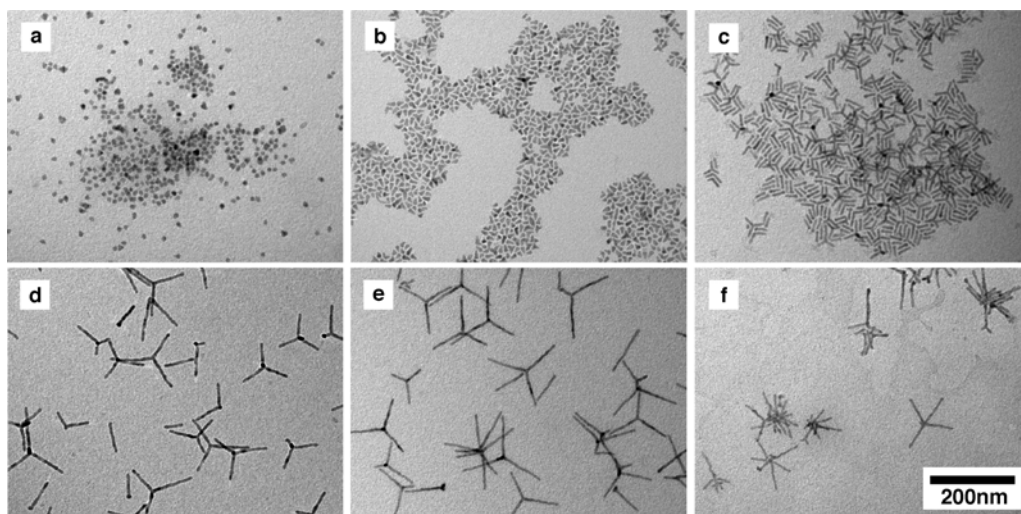
Fig. 1 Transmission electron microscopy (TEM) images of CdTe nanoparticles with different shape.

suppressed due to the better passivation of this facet. The second model describes tetrapods as completely grown in the hexagonal phase, as each couple of arms is interconnected through two twin boundaries to another wurtzite domain located in the core of the tetrapod. In this model the tetrapods core can be considered as an assembly of eight building blocks that are connected to each other through twin boundaries. In the literature on ZnO tetrapods this model is known as the octatwin model [53].

Which of the two mechanism is operative in II–VI nanocrystals is object of investigation in our groups. In the case of CdTe nanocrystals, we are now able to tune the various reaction conditions so that we can synthesize nanocrystals having a variety of different shapes, ranging from spheres to rods, to nanocrystals that are progressively more branched (see Fig. 1). This controlled synthesis has also shed a light on the mechanism of branching in nanocrystals [54].

In a more elaborate approach to nanocrystal synthesis, starting from shape controlled CdSe, CdS or CdTe particles, nanocrystals of other materials, having the same shape or at least a similar shape can be obtained. This is possible for instance through a cation exchange reaction [55], which is even reversible. One demonstrated example is the replacement of Cd^{2+} ions in CdSe rods by Ag^+ ions to form Ag_2Se nanoparticles.

4 Synthesis of hybrid materials

The combination of different inorganic materials in one particle is desirable, as the resulting new material exhibits the properties of both constituents. Inorganic colloidal particles can be connected with organic, biological molecules in a post-synthesis step. Here the key-lock principle is exploited by binding one type of molecule to the first particle and the complementary molecule to the second type of particles. This technique has been demonstrated for the formation of DNA-connected gold particles of different size [57, 58]. Unfortunately, these structures are very sensitive to environmental changes. DNA linkers are for example not stable against heat, as the double strand would break open. Biological linkers are typically also floppy. In order to get more stable and rigid hybrid materials it is preferable to epitaxially link different materials together.

The most symmetric example of inorganic hybrid materials is the core-shell structure. In this system one particle is embedded into a shell of a different material. For instance fluorescent semiconductor particles can be surrounded by a shell of a semiconducting material with a higher band gap. In the case of CdSe nanoparticles, the growth of a ZnS shell enhances the fluorescence properties of the material and the quantum yield can be increased by more than a tenfold [59].
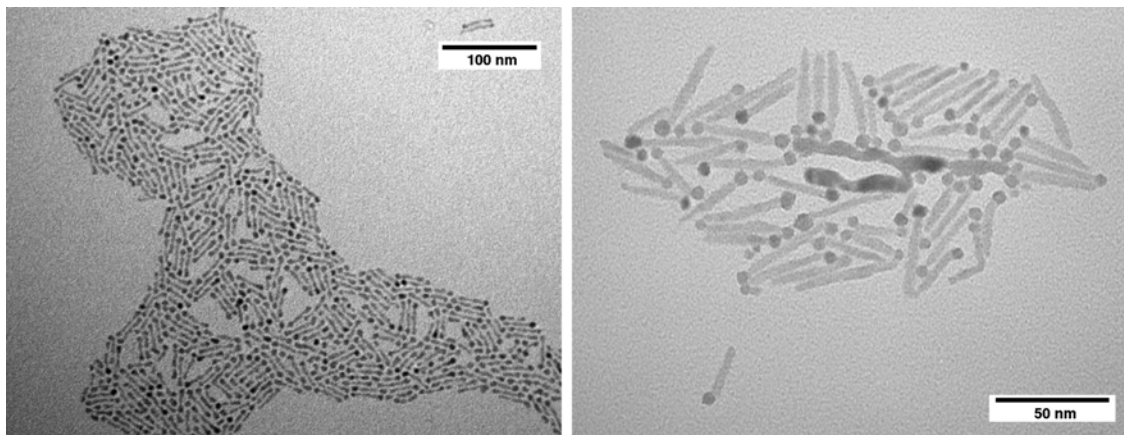
Fig. 2 TEM images of dumbbell and matchstick structures. The left images shows a sample of CdSe rods with PbSe spheres on their tips. The right image shows a sample of CdS rods with a PbSe sphere mainly only on one of their tips. All experimental procedures are reported in Ref. [60].

Other examples of heterostructures are based on nanorods. As discussed above, in the case of rod growth different facets of the crystals have different reactivities. The higher reactivity of the tips or the better passivation of the lateral facets give rise to the possibility to nucleate another material only on the tips of the rods. This scheme can be exploited to grow dumbbell-shaped structures. As an example recently reported by our groups [60] and by the group of Banin [61], first rods of CdSe or CdS can be synthesized and purified. Then, in the presence of these particles, the synthesis of a second material is performed. When the concentration of the monomers of the second material is sufficiently low, the new material nucleates only on the tips of the rods. It seems that stress that is provoked by the lattice mismatch between the two materials does not suppress the formation of such structures. Gold tipped CdSe rods [61] can be produced according to this technique as well as PbSe dots may be grown onto CdSe or CdS rods (see Fig. 2) [60]. In the later case, even the number of dots per particle can be controlled. Instead of dumbbells with two dots, matchsticks with only one dot can be synthesized. With a slightly different approach, ZnO–Ag heterostructures can be produced [62]. Also in this case a two step synthesis is necessary. First the ZnO nanorods are synthesized, then Ag is deposited onto these particles through a photocatalytic reaction. Upon strong UV irradiation, electron–hole pairs are generated in the ZnO rods, the electrons can then reduce the Ag-ions and so trigger the nucleation of Ag-particles on the surface of the rods.

In an *in-situ* approach it is possible to first synthesize rods of a cadmium-based semiconductor material and then grow from its tips linear sections of a different material (CdTe or CdSe). By carefully adjusting the experimental conditions also the shape of the central material (either rod or tetrapod) and the outer material (either linear or branched section) can be controlled [63].

A completely different approach to produce hybrid nanocrystals was followed by Gu and co-workers [64]. They reported the synthesis of hybrids with a CdS and a FePt section. Here, first an amorphous shell of CdS is grown onto FePt particles and then by annealing these samples, crystalline CdS domains form on one side of the FePt, due to a de-wetting process which minimizes the interfacial energy between the two materials.

5 Outlook

By carefully manipulating the thermodynamics and kinetics of crystal growth one can control both the shape and the composition of nanocrystals. It is now possible to grow several materials in complex shapes, such as for instance tetrapods. Starting from these structures, one can synthesize hybrid materials

by exploiting the different passivation efficiencies of surfactant molecules towards the various crystal facets.

These new materials are promising candidates for different applications. First, they might open new perspectives in self-assembly, as in the case of metal tipped semiconductor rods. Reactive groups such as thiols bind strongly to Au, but only relatively weakly to CdSe and CdS surfaces [61]. This facilitates site-selective surface functionalization by organic molecules. By simply exposing CdSe–Au dumbbell structures to biological molecules bearing free thiol-groups, selective attachment of these molecules at the gold-tip regions is expected. First demonstrations in this direction have been reported already for hybrid materials of bigger size [65], namely Au/Ni particles. It was demonstrated that different reactive groups bind selectively to certain domains of a nanocrystal. In particular, thiol-functionalised biomolecules were bound to the gold domains, carboxylic groups were bound to the Ni domain. Also for nanometer-sized CdSe–Au dumbbell structures size-selective surface functionalization has been reported [61]. By adding dithiols, CdSe–Au dumbbells are selectively connected at their Au-ends, which leads to the formation of nanorod chains [61]. Nevertheless, experiments in this direction are just in their infancy and still many questions, such as the role of non-specific interactions remain to be solved. Site-specific functionalization on just one tip of the rods would facilitate new possibilities of arranging particles on surfaces. For an example, by linking rod-shaped objects with their tip to a surface, a brush of vertically aligned particles could be formed (see Fig. 3). This structure would be of great interest for the fabrication of solar cells [5]. Certainly, the possibility to selectively functionalize specific regions of the particles with organic molecules will play an important role in the future for the generation of complex self-assembled structures with an improved degree of programmability.

Other possible applications of hybrid particles are expected in the field of electronics [14, 20]. Gold tipped nanocrystals may render it easier to form electronic contacts with the particles. This might for example help the design of nanocrystal-modified electrodes [66]. In addition, linear junctions of semiconductor particles may provide interesting electronic properties by themselves. The band structure of heterostructures of two types of semiconductor may open up new applications for this material. The reported PbSe–CdSe–PbSe heterostructure [60] could be considered as a light-controlled transistor. Here a high-band gap material (CdSe, $E_g \approx 2$ eV) is situated between two sections of a material with smaller band gap (PbSe, $E_g \approx 0.5$ eV). In this way, communication between the two PbSe sections only is possible when they are in an excited state, that is after the absorption of a photon.

All synthesis procedures and reactions reported so far are dependent on organic molecules bearing long alkyl chains (the surfactants) and therefore the as-grown nanoparticles are hydrophobic. Several methods are now available to transfer these particles to aqueous solution, which work by rendering the surface of these nanocrystals hydrophilic [29, 67]. Water-soluble particles offer a whole new direction of applications, especially in the field of life sciences. We refer here to recent review articles [31, 32, 68]. It is currently under discussion if nanoparticles are compatible with biological applications. There are several investigations on this subjects [69–71], but it seems that this question is not easy to address, as for

Fig. 3 Selective functionalization may be used for the formation of complex architecture as shown in a sketch here. By functionalizing only the tips of the dumbbell structures it may be possible to form chains of these particles (left sketch). This has been demonstrated already [61] by carefully exposing gold tipped nanorods to hexane(1,6)dithiol Providing functional pads (right sketch) on a substrate may enable the formation of brushes of rods. For this approach matchstick-shaped particles with only one functional domain could be of advantage.

instance in the case of gold-nanoparticle there is a strong, non-linear dependence of the toxicity on the size of the particles [72, 73]. Hybrid nanoparticles also open new perspectives in life science applications. Nanoparticles with combined magnetic and fluorescent properties [64] could be directed with magnetic field gradients and detected on a single-particle level by their fluorescence.

We believe that in future the synthesis of nanocrystals should evolve into the development of more complex nano-structures. An appealing field of research would be the synthesis of hybrid nano-materials with increased functionality.

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