

Review Article

The Progress of Photoluminescent Properties of Rare-Earth-Ions-Doped Phosphate One-Dimensional Nanocrystals

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One-dimensional (1D) nanostructures, such as tubes, wires, rods, and belts, have aroused remarkable attentions over the past decade due to a great deal of potential applications, such as data storage, advanced catalyst, and photoelectronic devices. On the other hand, in comparison with zero-dimensional (0D) nanostructures, the space anisotropy of 1D structures provided a better model system to study the dependence of electronic transport, optical and mechanical properties on size confinement and dimensionality. Rare earth (RE) compounds, were intensively applied in luminescent and display devices. It is expected that in nanosized RE compounds the luminescent quantum efficiency (QE) and display resolution could be improved. In this paper, we systematically reported the research progress of luminescent properties of RE-doped 1D orthophosphate nanocrystal, including the synthesis of 1D nanostructures doped with RE ions, local symmetry of host, electronic transition processes, energy transfer (ET), and so forth.

1. Introduction

It is well known that the reduction of particle size of crystalline system can result in remarkable modification of their properties which are different from those of microsized hosts because of surface effect and quantum confinement effect of nanometer materials. In 1994, Bhargava et al. reported that radiative transition rate of ZnS:Mn nanocrystals increased five orders in comparison with bulk one [1]. Although this result was strongly criticized later, the studies on nanosized luminescent semiconductor attracted great interests [2–6]. RE compounds were extensively applied in luminescence and display, such as lighting, field emission display (FED), cathode ray tubes (CRT), and plasma display panel (PDP) [7–11]. Lanthanide orthophosphate (LnPO_4) belongs to two polymorphic types, the monoclinic monazite type (for La to Gd) and quadratic xenotime type (for Tb to Lu). Due to its high QE, bulk lanthanide phosphate as an ideal host in fluorescent lamps, CRT and PDP, has been extensively investigated [12–14]. It is expected that nanosized RE compounds can increase luminescent QE and display resolution. To improve

luminescent properties of nanocrystalline phosphors, many preparation methods have been used, such as solid state reactions, sol-gel techniques, hydroxide precipitation, hydrothermal synthesis, spray pyrolysis, laser-heated evaporation, and combustion synthesis. Currently, the luminescent RE-doped 1D nanocrystals such as LaPO_4 :RE nanowires [15–19], Y_2O_3 :RE and La_2O_3 :Eu nanotubes/nanowires [20–25], and YVO_4 :Eu nanowires/nanorods [26–28]. have also attracted considerable interests. 1D structures, such as tubes, wires, rods, and belts, have aroused remarkable attentions over past decade due to a great deal of potential applications, such as data storage [29], advanced catalyst [30], and photoelectronic devices [31]. On the other hand, in comparison with 0D structures, the space anisotropy of 1D structures provided a better model system to study the dependence of electronic transport and optical and mechanical properties on size confinement and dimensionality [32]. To develop 1D phosphors, a basic question should be answered: could the photoluminescent properties for 1D nanocrystals be improved than 0D ones as well as the micrometer materials, the so-called bulk materials? In this review, we concentrated

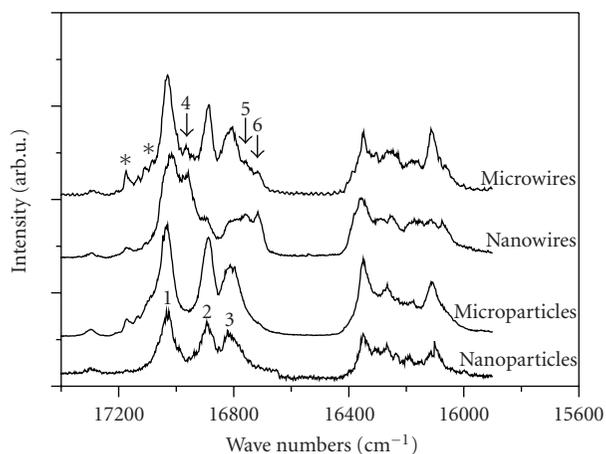


FIGURE 1: High-resolution spectra of $\text{LaPO}_4:\text{Eu}^{3+}$ bulk, nanoparticles, and nanowires at 10 K under 266 nm light excitation (delay time is 50 μs). (J. Phys. Chem. B 2004, 108, 16697; Appl. Phys. Lett. 2004, 85, 470).

on the study progress of photoluminescent properties of RE ions in phosphate 1D nanocrystals.

2. The Synthesis of Phosphate 1D Nanocrystals Doped with RE

In 1999, Meyssamy et al. reported the preparation of $\text{LaPO}_4:\text{Eu}^{3+}/\text{Tb}^{3+}$ nanowires by a hydrothermal method and studied their luminescent properties for the first time [15]. After then, researchers prepared phosphate 1D nanocrystals doped with RE ions by different synthesis techniques, such as hydrothermal method to prepare $\text{LaPO}_4:\text{Ce}^{3+}/\text{Eu}^{3+}$ nanofibers [33], $\text{CePO}_4:\text{Tb}^{3+}$ colloidal nanocrystals [34], $\text{CePO}_4:\text{Tb}^{3+}/\text{LaPO}_4$ nanowires core-shell structures [35, 36], and LnPO_4 ($\text{Ln} = \text{RE}$) nanowires and nanosheets [19]. It should be noted that these reports mainly emphasize the morphology control through changing synthesis conditions. To our knowledge, although LnPO_4 1D nanocrystals doped with RE ions with different shapes were prepared by the wet chemistry method by many groups, the luminescent properties, especially electronic transition processes and ET processes, were not further studied. Our group systemically reported luminescent characteristics of $\text{LnPO}_4:\text{RE}$ ($\text{Ln} = \text{La}, \text{Gd}$) nanowires and nanorods. Song and Yu et al. systemically and further investigated the $\text{LaPO}_4:\text{Eu}^{3+}/\text{Tb}^{3+}$ nanowires photoluminescent properties in comparison with 0D nanoparticles and bulk materials, especial electronic transition process and ET.

3. Local Symmetry of Eu^{3+} in 1D Nanocrystals

Because Eu^{3+} ions were hypersensitive to local structures, they can be used as a fluorescent probe to detect the microstructures surrounding Eu^{3+} ions. In previous research, the local environmental around Eu^{3+} ions in 0D nanoparticles was systematically studied. Many groups reported the appearance of additional sites of Eu^{3+} ions in Y_2O_3 and

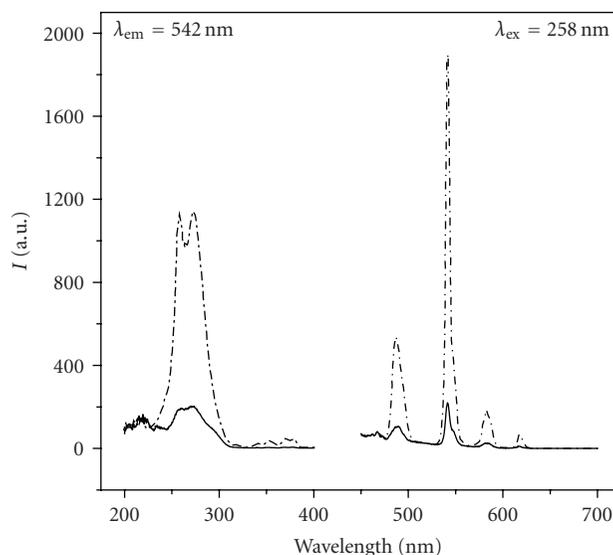


FIGURE 2: The excitation spectra (left) and emission (right) for $\text{LaPO}_4:1\%\text{Ce}, 2.5\%\text{Tb}$ samples. Solid lines and dash dots represented the microrods and nanowires, respectively. (J. Phys. Chem. B 2005, 109, 11450).

YBO_3 nanoparticles through high-resolution spectra at low temperature [37–40]. They contributed the origin of additional sites of Eu^{3+} doped nanoparticles to the surface effect because the disorder of atoms at the surface of nanoparticles increased on comparison with the inside atoms. Although $\text{LnPO}_4:\text{Eu}^{3+}$ 1D nanowires/nanorods were prepared and their basic spectra were researched, the dependence of local symmetry on shape was not studied. Song and Yu et al. investigated the microstructures of Eu^{3+} doped 1D LaPO_4 nanowires in contrast with 0D nanoparticles and corresponding micrometer hosts (micrometer particles and rods) [16, 18, 41]. The LaPO_4 nanoparticles, nanowires, and bulk materials were synthesized by the same synthesis technique, a hydrothermal method. All samples were at monazite phase and no additional phases were observed. The size of $\text{LaPO}_4:\text{Eu}^{3+}$ nanoparticles is ranging from 10 to 20 nm. The diameter of $\text{LaPO}_4:\text{Eu}^{3+}$ nanowires ranges from 10 to 20 nm, and the length is about several hundreds nanometer. They observed that the Eu^{3+} ions locate new site in nanowires due to shape effect. Figure 1 shows the high-resolution spectra of $\text{LaPO}_4:\text{Eu}^{3+}$ nanomaterials and bulk materials at 266 nm pulsed laser excitation at 10 K. The emission associated with $^5\text{D}_0-^7\text{F}_1$ transitions is quite different between nanoparticles and nanowires at 10 K. In the nanoparticles, three $^5\text{D}_0-^7\text{F}_1$ emission lines were observed, locating at $17025 \pm 2 \text{ cm}^{-1}$ (L1), $16898 \pm 2 \text{ cm}^{-1}$ (L2), and $16815 \pm 2 \text{ cm}^{-1}$ (L3), respectively. In the nanowires, besides the same lines 1–3, three additional lines 4–6 were observed, locating at $16963 \pm 2 \text{ cm}^{-1}$ (L4), $16758 \pm 2 \text{ cm}^{-1}$ (L5), and $16718 \pm 2 \text{ cm}^{-1}$ (L6), respectively. The $^5\text{D}_0-^7\text{F}_1$ lines in the microparticles are identical with the nanoparticles. For the microrods, lines 1–6 were also observed. However, the relative intensity of lines 4–6 becomes weaker in comparison with that in nanowires. $^7\text{F}_1$ associated with one site symmetry can split into three

TABLE 1: A list of parameters W_1 , $W_{10}(0)$ and the internal luminescent QE at 0 K in different powders.

Parameters	nanoparticles	nanowires	microparticles	microrods
W_1 (ms ⁻¹)	14.9	28.9	17.6	16.5
W_{10} (ms ⁻¹)	24.1	19.7	17.8	18.5
QE	38%	59%	49%	47%

Stark lines in the crystal field. The results in Figure 1 indicate that in nanoparticles and microrods, the 5D_0 - 7F_1 transitions are from one crystalline site, A, while in nanowires and microrods, the 5D_0 - 7F_1 transitions come from the same site (L1-L3), A, and an additional site (L4-L6), B. The relative number of Eu^{3+} at site B decreases as the powders vary from the nanowires to the microrods. In the present case, from the microparticles to the nanoparticles, the ratio of surface to volume increases greatly, but no additional site is observed. From the microrods to the nanowires, the ratio of surface to volume do not increase so much; however, the additional site B appears and the relative number of Eu^{3+} at site B changes greatly. We thus believe that the appearance of the additional site B is not caused by the surface effect, but by the shape anisotropy. This is the first time to report that the shape effect affects the local structures of Eu^{3+} doped 1D nanocrystals.

4. Electronic Transition Processes and QE of Eu^{3+} in LaPO_4 1D Nanocrystals

In 0D nanoparticles, many groups reported that the QE and luminescent intensity of Eu^{3+} ions decreased in comparison with the bulk materials due to higher nonradiative transition rate in nanoparticles [42, 43]. An important problem should be answered: does the QE of Eu^{3+} in 1D nanocrystals increase in contrast with nanoparticles and bulk materials? Song and Yu et al. systematically studied the electronic transition processes of Eu^{3+} in LaPO_4 nanowires [16, 18, 41]. The luminescence QE of 5D_1 level of Eu^{3+} was determined by the following equation:

$$\tau(T) = \frac{1}{W_1 + W_{10}(T)}, \quad (1)$$

where W_1 is the radiative transition rate of $^5D_1 - \sum_j ^7F_j$, $W_{10}(T)$ is nonradiative transition rate at a certain temperature, T . According to the theory of multiphonon relaxation, W_{10} can be written as

$$W_{10}(T) = W_{10}(0)(1 + \langle n \rangle)^{\Delta E_{10}/\hbar\omega}, \quad (2)$$

where $W_{10}(0)$ is nonradiative transition rate at 0 K, ΔE_{10} is the energy separation between 5D_1 and 5D_0 , $\hbar\omega$ is the phonon energy, k is Boltzmann' constant, and $\langle n \rangle = 1/(e^{\hbar\omega/kT} - 1)$ is the phonon occupation number. According to (1) and (2), the lifetime of 5D_1 can be expressed as

$$\tau = \frac{1}{W_1 + W_{10}(0)[1 - \exp(-\hbar\omega/kT)]^{-\Delta E_{10}/\hbar\omega}}. \quad (3)$$

They measured the lifetime of 5D_1 level of Eu^{3+} ions at different temperatures and obtained the radiative and

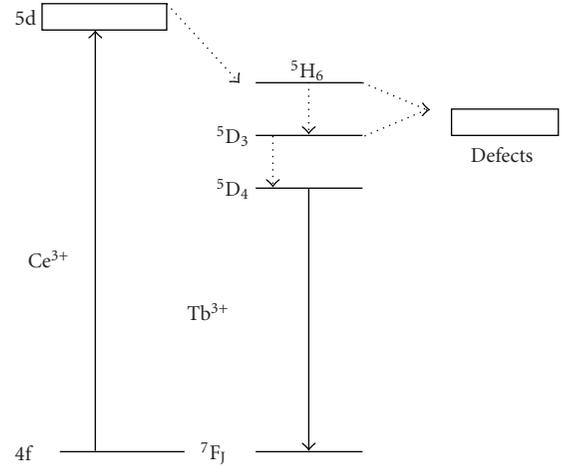


FIGURE 3: Schematic for the ET and luminescence processes in $\text{Ce}^{3+}/\text{Tb}^{3+}$ codoped LaPO_4 nanowires and microrods. (J. Phys. Chem. B 2005, 109, 11450).

nonradiative transition rate by fitting according to (3). The results were listed in Table 1. It is obvious that the radiative transition rate and QE of 5D_1 level in nanowires were higher than that in nanoparticles and bulk crystals. This result indicates that the 1D nanocrystal-doped RE ions is ideal phosphors. After then, they also studied the electronic transition processes of Eu^{3+} ions in Y_2O_3 and La_2O_3 nanowires [24, 25]. The results indicate that the radiative transition rate of Eu^{3+} in oxide nanowires hardly changes in comparison with the bulk hosts.

5. ET between RE Ions in LaPO_4 1D Nanowires

Ce^{3+} and Tb^{3+} ions are important RE ions, which have been applied in blue and green phosphors. The ET processes between Ce^{3+} and Tb^{3+} in some micrometer-sized materials, such as lanthanum oxybromide [44], aluminate [45], alkaline earth sulphate [46], and so on, were intensively investigated. As efficient green phosphors, Ce^{3+} and Tb^{3+} coactivated LaPO_4 bulk powders were extensively applied to fluorescent lamps, cathode ray tube (CRT), and plasma display panel (PDP) due to the high ET efficiency between Ce^{3+} and Tb^{3+} ions [13]. To obtain the efficient green phosphors of 1D $\text{LaPO}_4:\text{Ce}^{3+}/\text{Tb}^{3+}$ nanowires, the electronic transition and ET processes in 1D nanowires should be studied, and compared with the corresponding bulk powders. However, the studies on ET processes between Ce^{3+} and Tb^{3+} , even between different RE impurity centers, are rather rare. Song and Yu et al. fabricated Ce^{3+} and Tb^{3+} coactivated LaPO_4 nanowires as well as the micrometer-sized rods by the same hydrothermal method [17, 47]. They systematically studied and compared their electronic transition and ET processes by the luminescent spectra and dynamics analysis. Figure 2 shows the excitation and emission spectra of $\text{LaPO}_4:\text{Ce}^{3+},\text{Tb}^{3+}$ nanowires and microrods. It is obvious and important that the luminescent intensity of Tb^{3+} in $\text{LaPO}_4:\text{Ce}^{3+},\text{Tb}^{3+}$ nanowires is much stronger than that in

TABLE 2: List of dynamic results of $\text{LaPO}_4:0.01\text{Ce}^{3+}, 0.025\text{Tb}^{3+}$ nanowires and microrods.

Samples	Electronic transition rate of Ce^{3+} (ns^{-1})	Lifetime of Tb^{3+} (ms)	ET ($\text{ns}^{-1}\text{mol}^{-1}$)
nanowires	6.3×10^{-2}	2.72	0.44
microrods	5.2×10^{-2}	2.58	1.30

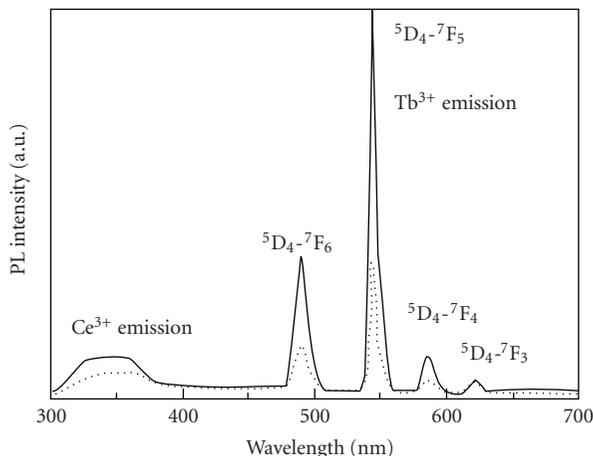


FIGURE 4: Fig. 4 Luminescence emission ($\lambda_{\text{ex}} = 278 \text{ nm}$) spectra of the dilute colloidal solutions of the $\text{Ce}_{0.9}\text{Tb}_{0.1}\text{PO}_4$ 1D nanostructures (dashed line) and the $\text{Ce}_{0.9}\text{Tb}_{0.1}\text{PO}_4@ \text{LaPO}_4$ 1D nanocable heterostructures (solid). (J. Phys. Chem. B 2008, 109, 14461).

microrods corresponding to the excitation of Ce^{3+} ions. It should be noted that the emission of Ce^{3+} or Tb^{3+} in single-doped nanowires is lower than that in microrods. The dynamic results were listed in Table 2. The dynamic measurements and analysis indicate that the electronic transition rate of Ce^{3+} and Tb^{3+} , the ET rate from Ce^{3+} to Tb^{3+} in Ce^{3+} - Tb^{3+} codoped LaPO_4 nanowires, is lower than that in microrods. They attributed the increased intensity of Tb^{3+} in nanowires to the lower ET efficiency from Tb^{3+} to other quench centers, shown in Figure 3. According to the Eu^{3+} and Tb^{3+} doped LaPO_4 nanowires, we suggested that the RE-doped 1D nanowires were ideal nanophosphors. In addition, we also observed the ET from Gd^{3+} to Tb^{3+} in $\text{GdPO}_4:\text{Tb}^{3+}$ nanorods [48].

6. The Core Shell Nanostructures of LaPO_4 Nanowires Doped with RE

Due to the increased disorder of surface atoms for nanomaterials, the nonradiative transition rate of RE-doped nanocrystals will increase. The surface modification can effectively eliminate the quenching centers at surface of nanocrystal. In 1999, Li et al prepared $\text{Y}_2\text{O}_3:\text{Eu}^{3+}@ \text{SiO}_2/\text{Al}_2\text{O}_3$ nanoparticles heterostructures and observed the increase of Eu^{3+} luminescence [49]. In 2003, Haase prepared the $\text{CePO}_4:\text{Tb}^{3+}/\text{Eu}^{3+}@ \text{LaPO}_4$ nanoparticles core-shell structures and observed that the quantum yield of Tb^{3+} was increased from 43% to 70% [50, 51]. Lin group systematically studied 0D nanoparticles doping with RE coated with SiO_2 [52–67]. Recently, 1D nanosized core-shell structures

doped with RE have been reported. Bu et al. reported uniform $\text{CePO}_4@ \text{LaPO}_4$ and $\text{CePO}_4:\text{Tb}^{3+}@ \text{LaPO}_4$ 1D single-crystalline nanocable heterostructures with highly enhanced photoluminescent emission [36]. The resulting 1D single-crystalline nanocable heterostructures have smooth and uniform LaPO_4 sheaths, which is of great significance in effectively eliminating surface trap-states and suppressing the energy quenching in ET processes. The photoluminescence results for these 1D nanocable heterostructures illustrate that the uniform LaPO_4 sheaths remarkably increase the luminescent efficiency. Figure 4 shows the emission spectra of ($\lambda_{\text{ex}} = 278 \text{ nm}$) the dilute colloidal solutions of the $\text{Ce}_{0.9}\text{Tb}_{0.1}\text{PO}_4$ 1D nanostructures (dashed line) and the $\text{Ce}_{0.9}\text{Tb}_{0.1}\text{PO}_4@ \text{LaPO}_4$ 1D nanocable heterostructures (solid lines). It is obvious that the emission intensity of Tb^{3+} and Ce^{3+} in $\text{LaPO}_4:\text{Ce}^{3+}, \text{Tb}^{3+}$ nanowires coated with LaPO_4 is much high than that in noncoated samples.

Fang et al. also reported that $\text{CePO}_4:\text{Tb}^{3+}/\text{LaPO}_4$ core/shell nanowires synthesized by a simple hydrothermal method, and the resulting 1D core/shell nanostructures have high photoluminescence efficiency [35]. Until recently, it has been considered difficult to obtain a nanocrystal phosphor material with the quantum yield being close to that of the corresponding bulk material. The novel core/shell nanostructures, which are more robust than organically passivated nanowires, may be used as building blocks for optoelectronic nanodevice applications. $\text{CePO}_4:\text{Tb}/\text{LaPO}_4$ core/shell nanowires with high quantum yield would also be a new class of biomedical labels for ultrasensitive, multicolor, and multiplexing applications owing to their nontoxicity and biocompatibility.

In summary, we introduced the recent progresses of phosphate 1D nanowires doped with RE. We suggest that the research focus should be the application of 1D nanowires in biological probe, photonic crystals, optical communication, etc in future.

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