

## Research Article

# The Influence of SiO<sub>2</sub> Shell on Fluorescent Properties of LaF<sub>3</sub>:Nd<sup>3+</sup>/SiO<sub>2</sub> Core/Shell Nanoparticles

Cui Kai,<sup>1,2</sup> Gao Chao,<sup>1</sup> Peng Bo,<sup>1,3</sup> and Wei Wei<sup>3</sup>

<sup>1</sup> State Key Laboratory of Transient Optics and Photonics, Xi'an Institute of Optics and Precision Mechanics, Chinese Academy of Science (CAS), Xi'an Shaanxi 710119, China

<sup>2</sup> Graduate School of the Chinese Academy of Sciences, Beijing 100039, China

<sup>3</sup> Institute of Advanced Materials, Nanjing University of Posts and Telecommunications, Nanjing 210003, China

Correspondence should be addressed to Peng Bo, bpeng@opt.ac.cn

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Distinct effects of the SiO<sub>2</sub> shell on fluorescence properties of LaF<sub>3</sub>:Nd<sup>3+</sup>/SiO<sub>2</sub> core/shell nanoparticles were demonstrated by annealing the nanoparticles at different temperatures. Emission spectra, excitation spectra, and decay curves of the nanoparticles were measured. A significant improvement of fluorescence intensity was observed for LaF<sub>3</sub>:Nd<sup>3+</sup>/SiO<sub>2</sub> core/shell nanoparticles annealed at 900°C. The phenomenon is ascribed to the change of environment of LaF<sub>3</sub>:Nd<sup>3+</sup> core which is imposed by SiO<sub>2</sub> shell. And the change is confirmed by the excitation spectra. It provides a useful way to improve fluorescent intensity of the SiO<sub>2</sub>-coated LaF<sub>3</sub>:Nd<sup>3+</sup> nanoparticles. The lifetime for nanoparticles annealed at 900°C shows a slight decrease contrast with nanoparticles annealed at 400 and 600°C. This is caused by the higher phonon energy of SiO<sub>2</sub> than that of LaF<sub>3</sub>.

## 1. Introduction

In the past decade, the synthesis of lanthanide-doped nanoparticles has attracted a great deal of attention, since the materials are considered as potentially useful phosphors in lamps, display devices [1], components in optical telecommunication [2], new optoelectronic devices [3], and probes in biomedical imaging and detection [4]. LaF<sub>3</sub> possessing low phonon energy, adequate thermal and environmental stability, is regarded as excellent host matrixes for performing luminescence [5, 6]. Nanoparticles of LaF<sub>3</sub> doped with lanthanide ions have been studied for years for their luminescence properties [7, 8]. However, the water and organic molecules absorbed on nanoparticles noticeably hampered their optical efficiency, when the nanoparticles are dispersed into aqueous and organic environment. The O-H and C-H groups have a high vibration frequency and can efficiently quench the luminescence of lanthanide ions [9, 10]. This is in particular true for the lanthanide ions emitted in the near-infrared region, like Nd<sup>3+</sup>, Yb<sup>3+</sup>, and Er<sup>3+</sup> because the energy gap between excited state and ground state is small [11]. Fortunately, these problems can

be overcome when an appropriate shell is grown around the lanthanide ions doped LaF<sub>3</sub> core, and silica is usually used as a coating layer due to its high chemical stability, optical transparency, and biocompatibility [12]. LaF<sub>3</sub> nanoparticles with different thickness of SiO<sub>2</sub> shell were synthesized and the LaF<sub>3</sub>:Nd<sup>3+</sup>/SiO<sub>2</sub> core/shell nanomaterials used for biological NIR probes has been reported [13, 14]. However, except the protection effect of SiO<sub>2</sub> layer, the influence of SiO<sub>2</sub> shell on fluorescent properties of the Lanthanide-doped LaF<sub>3</sub> core has seldom been discussed.

In this work, To investigate the interactions between SiO<sub>2</sub> shell and lanthanide ions doped LaF<sub>3</sub> core, a series of Nd<sup>3+</sup>-doped LaF<sub>3</sub> nanoparticles capped with SiO<sub>2</sub> shell were synthesized and annealed at different temperatures. When the anneal temperature is 900°C, spectroscopic evidence for the change of LaF<sub>3</sub> environment created by SiO<sub>2</sub> shell was observed. And the change of environment leads to a significant improvement of the fluorescent intensity of LaF<sub>3</sub>:Nd<sup>3+</sup>/SiO<sub>2</sub> core/shell nanoparticles. This provides a simple and useful way to improve the fluorescent properties of lanthanide-doped LaF<sub>3</sub>/SiO<sub>2</sub> core/shell nanomaterials.

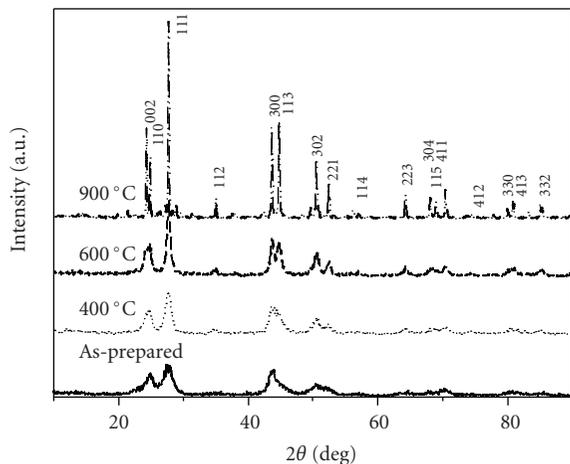


FIGURE 1: XRD patterns of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles annealed at different temperatures.

## 2. Experimental

The  $\text{SiO}_2$ -coated  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles were synthesized as follows.  $\text{NH}_4\text{F}$  (0.44 g, 12 mmol) was dissolved in methanol (20 mL), and then the solution was heated to  $65^\circ\text{C}$ . Another solution of  $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  (1.694 g, 3.89 mmol),  $\text{Nd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  (0.052 g, 0.11 mmol) in methanol (10 mL) was added dropwise to the  $\text{NH}_4\text{F}$  solution. The resulting solution was stirred at  $65^\circ\text{C}$  for 2 h. And the  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles were collected by centrifugation. Stöber method was adopted for the  $\text{SiO}_2$  coating process.  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles (0.5 g) were dispersed in ethanol (100 mL). Then tetraethyl orthosilicate (TEOS) (0.2 mL, 1 mmol) was added dropwise to the solution. After mixing for 1 min,  $\text{NH}_4\text{OH}$  (25%) (3 mL) were added in the mixture under stirring. The mixture was stirred for 3 h to get the as-grown sample. For better crystallinity and enhanced luminescence, the as-grown sample was annealed in the air for 4 h to get the final product.

The Inductively coupled plasma (ICP) analyses were carried out on a Hitachi P-4010 inductively coupled plasma emission spectrometer. X-ray diffraction (XRD) patterns were measured on a Rigaku D/max-2400 X-ray powder diffractometer. The size and morphology of nanoparticles were determined at 300 kV by a JEOL JEM-3010 transmission electron microscope (TEM) and XL30 field-emission scanning electron microscope (SEM). Photoluminescence emission spectra were recorded on a Zolix Omini-k 300 spectrophotometer pumped by a laser diode at 800 nm. The Fourier transform infrared (FTIR) spectra were made with a Shimadzu FT-IR 8900 spectrometer. The excitation spectra were recorded on an Edinburgh Instruments FLS920 spectrofluorimeter.

## 3. Results and Discussion

The concentrations of Nd, La, and Si in  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles were determined to be 1.79, 57.1, and 7.93% by ICP. The XRD patterns of samples annealed

at different temperatures are shown in Figure 1. When the as-prepared sample was heated at  $900^\circ\text{C}$  for 4 h, well-defined diffraction peaks were obtained. All the peaks can be well indexed to the hexagonal  $\text{LaF}_3$  crystal structure, and no trace of other characteristic peaks were observed. The TEM and SEM images provide direct information about the sizes and typical shapes of the nanoparticles. Figure 2 illustrates the representative TEM and SEM images of the  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles and those coated of  $\text{SiO}_2$  shells. The bare  $\text{LaF}_3:\text{Nd}^{3+}$  sample contains nanoparticles with an average size of 8 nm. After coating the  $\text{SiO}_2$  shell, it is clearly observed that the particles have a core-shell structure and the silica shell thickness is about 5–7 nm. When the  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  nanoparticles were annealed at  $900^\circ\text{C}$  for 4 h, the morphology of the sample aggregates with a size from 30–60 nm, and the SEM image (Figure 2(d)) showed that the annealed  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  nanoparticles consists of spherical particles with a size between 50–150 nm.

The FTIR spectra of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles are presented in Figure 3. Strong vibrational absorption bands at 3400–3600 and 1350–1600  $\text{cm}^{-1}$  were observed in as-prepared sample, which correspond to O-H mode. So the physically adsorbed solvent and O-H groups on the as-prepared nanoparticles are still not removed. Whereas for the nanoparticles annealed at  $400^\circ\text{C}$ , the former absorption peaks show a great decrease. When the anneal temperature raised to  $900^\circ\text{C}$ , the absorption peaks of O-H mode completely disappeared. Thus, the nonradiative vibrational excitation of  $\text{Nd}^{3+}$  in the nanoparticles created by O-H and C-H groups can be excluded.

Figure 4 shows the room temperature emission spectra of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell and  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles under excitation at 808 nm. The emission lines centered at 880, 1060, and 1330 nm correspond to the transitions from  $^4\text{F}_{3/2}$  to  $^4\text{I}_{9/2}$ ,  $^4\text{I}_{11/2}$ , and  $^4\text{I}_{13/2}$ , respectively [15]. For  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles as-prepared and annealed at 400 and  $600^\circ\text{C}$ , the emission pattern is similar with that of  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles in both the peak positions and shapes, which means that the  $\text{SiO}_2$  shell have minimal effect on  $\text{LaF}_3:\text{Nd}^{3+}$  core. However, when the annealed temperature is  $900^\circ\text{C}$ , emission spectra of the  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles show a very unusual manner. (1) Their fluorescence intensity show a great increase compared with that of the  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles annealed at  $600^\circ\text{C}$ . (2) The strongest emission line for  $^4\text{F}_{3/2} \rightarrow ^4\text{I}_{11/2}$  transition peaking at 1073 nm are redshifted by about 17 nm contrasted with that of nanoparticles annealed at  $600^\circ\text{C}$  (1056 nm). (3) The  $^4\text{F}_{3/2} \rightarrow ^4\text{I}_{9/2}$  and  $^4\text{F}_{3/2} \rightarrow ^4\text{I}_{13/2}$  emission of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles annealed at  $900^\circ\text{C}$  have remarkable Stark splittings which have not been found in other samples. In contrast, emission spectrum of  $900^\circ\text{C}$  annealed  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles which have no  $\text{SiO}_2$  shell has also been recorded, and is shown in Figure 4(b). The fluorescence intensity is only about 1/5 compared with that of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles annealed at  $900^\circ\text{C}$ , the  $^4\text{F}_{3/2} \rightarrow ^4\text{I}_{11/2}$  emission is peaking at about 1058 nm, and no emission lines have Stark splittings. The results mean, for  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles, that the influence

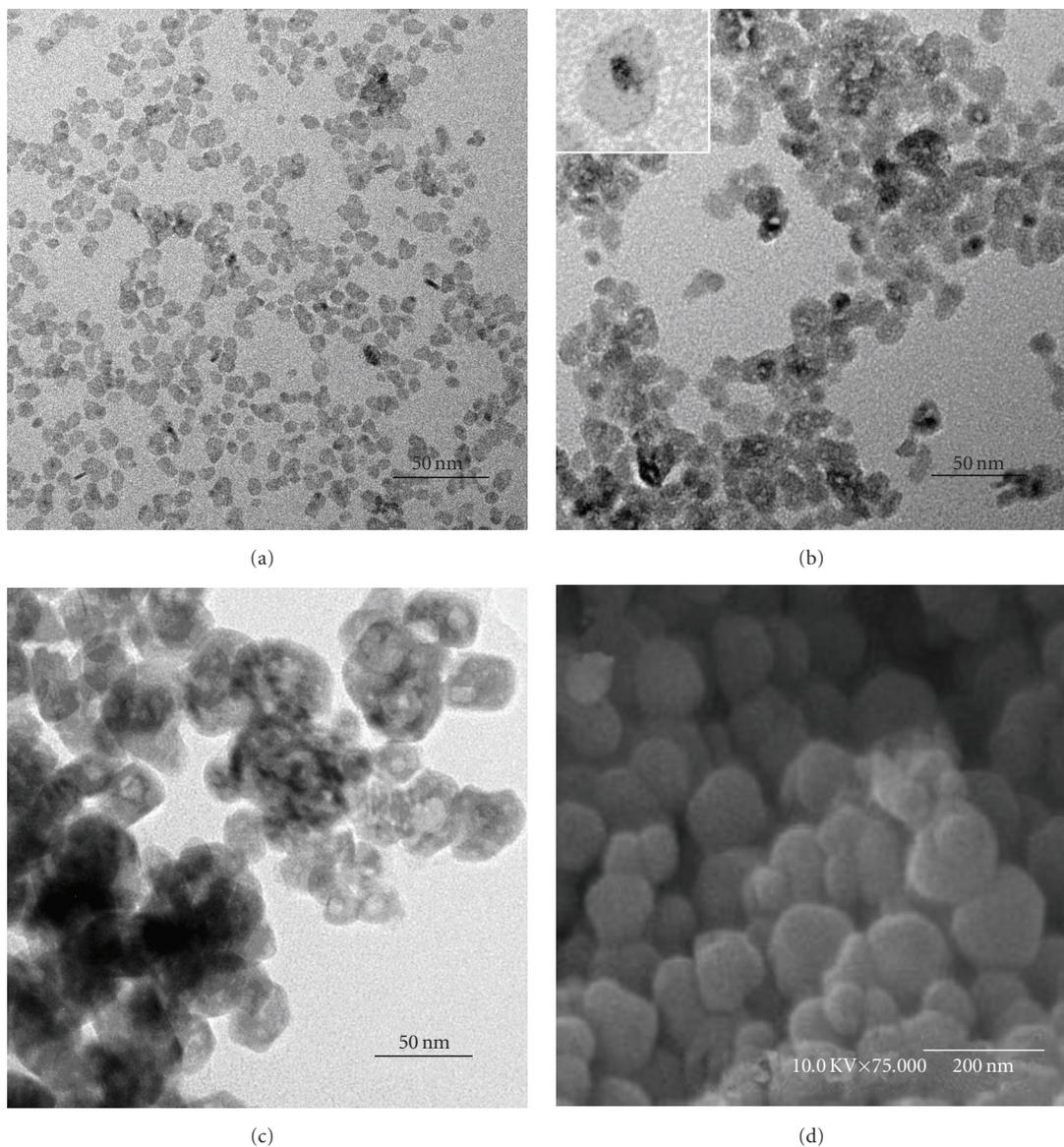


FIGURE 2: TEM images of (a)  $\text{LaF}_3:\text{Nd}^{3+}$ , (b)  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$ , (c)  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  annealed at  $900^\circ\text{C}$ , SEM image of (d)  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  annealed at  $900^\circ\text{C}$ .

of  $\text{SiO}_2$  shell has become clearly when the anneal temperature is  $900^\circ\text{C}$ , and the remarkable improvement of fluorescence intensity is caused by the  $\text{SiO}_2$  shell.

To get more information on the functions of  $\text{SiO}_2$  shell, the excitation spectra by monitoring the  ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{11/2}$  emission in  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell and  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles annealed at  $900^\circ\text{C}$  are compared in Figure 5. The spectra displays well-resolved lines, centered at 328, 352, 430, 472, 519, 578.9, 737, 801, and 881 nm corresponding to the direct excitation of  $\text{Nd}^{3+}$  from  ${}^4\text{I}_{9/2}$  to the higher excited states:  ${}^2\text{D}_{5/2}$ ,  ${}^2\text{P}_{1/2}$ ,  ${}^4\text{G}_{11/2}$ ,  ${}^2\text{K}_{15/2} + {}^2\text{D}_{3/2} + {}^2\text{G}_{9/2}$ ,  ${}^2\text{K}_{13/2} + {}^4\text{G}_{7/2} + {}^4\text{G}_{9/2}$ ,  ${}^2\text{G}_{7/2} + {}^2\text{G}_{5/2}$ ,  ${}^2\text{H}_{11/2}$ ,  ${}^4\text{F}_{9/2}$ ,  ${}^4\text{S}_{3/2} + {}^4\text{F}_{7/2}$ ,  ${}^4\text{F}_{5/2} + {}^2\text{H}_{9/2}$ , and  ${}^4\text{F}_{3/2}$ . Interestingly, remarkable differences are observed in excitation spectra of the two samples. As shown in the inset of Figure 5, the shape of peaks for  ${}^4\text{I}_{9/2} \rightarrow {}^2\text{P}_{1/2}$  transition are different between the two

nanoparticles, and the lines for  ${}^4\text{I}_{9/2} \rightarrow {}^4\text{S}_{3/2} + {}^4\text{F}_{7/2}$ , and  ${}^4\text{F}_{5/2} + {}^2\text{H}_{9/2}$  transitions of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles are much broader compared with that of  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles. The changes of excitation spectra mean that the environment of  $\text{LaF}_3:\text{Nd}^{3+}$  is different for the two nanoparticles. The Stark splittings of  ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{9/2}$  and  ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{13/2}$  emission for  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles annealed at  $900^\circ\text{C}$  also confirmed the change. In previous reports, the phenomena were often caused by the host matrices or surfactants [15]. In this work, for  $\text{SiO}_2$  shell and  $\text{LaF}_3$  core have not the same lattice structures, the shell could bring a noncentrosymmetric environment of the  $\text{LaF}_3:\text{Nd}^{3+}$  core [16]. And the new noncentrosymmetric environment causes a significant improvement of fluorescent intensity of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles [17].

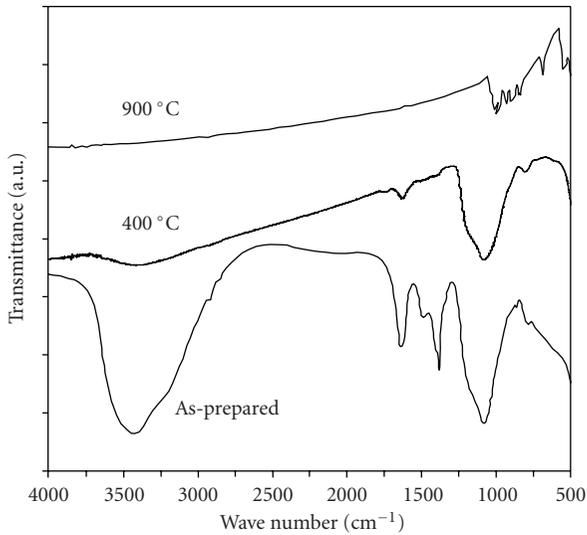


FIGURE 3: FTIR spectra of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles annealed at different temperatures.

TABLE 1: Lifetimes of nanoparticles annealed at different temperatures.

Nanoparticles	Annealed temperature ( $^{\circ}\text{C}$ )	Lifetime ( $\mu\text{s}$ )
$\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$ core/shell	400	159
	600	156
	900	148
$\text{LaF}_3:\text{Nd}^{3+}$	900	197

To have a further investigation of influence of the  $\text{SiO}_2$  shell on  $\text{LaF}_3:\text{Nd}^{3+}$  core, fluorescent decay curves of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles were measured by monitoring the  ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{11/2}$  emission at about 1060 nm. As shown in Table 1. For as-prepared nanoparticles, the lifetime is very difficult to be detected due to the presence of a large amount of O-H groups. When the nanoparticles were annealed at  $400^{\circ}\text{C}$ , the lifetime is  $159\ \mu\text{s}$ . Surprisingly, with a further rising of the anneal temperature, the lifetimes show a slight decrease. When annealed at 600 and  $900^{\circ}\text{C}$ , the lifetimes are  $156\ \mu\text{s}$  and  $148\ \mu\text{s}$ , respectively. However, for  $900^{\circ}\text{C}$  annealed  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles which have no  $\text{SiO}_2$  shell, the lifetime is  $196\ \mu\text{s}$ . It can be deduced that the lifetimes decrease in  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles is not caused by the  $\text{LaF}_3:\text{Nd}^{3+}$  core itself. In general, there are two factors that could affect radiative lifetime of  $\text{Nd}^{3+}$  in nanoparticles. (1) The existence of O-H groups and impurity on nanoparticles surfaces which could effectively reduce the lifetime of  $\text{Nd}^{3+}$ ; (2) vibrational energies of host matrix. For our samples, the difference is just the anneal temperature. For  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles annealed at  $900^{\circ}\text{C}$ , the effect of O-H groups can be obviated (Figure 3) and the  $\text{LaF}_3$  host has a good crystallinity (Figure 1).

The  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles were synthesized in two steps. In as-prepared nanoparticles,  $\text{SiO}_2$  shell

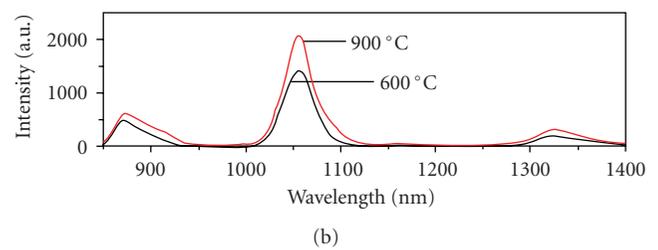
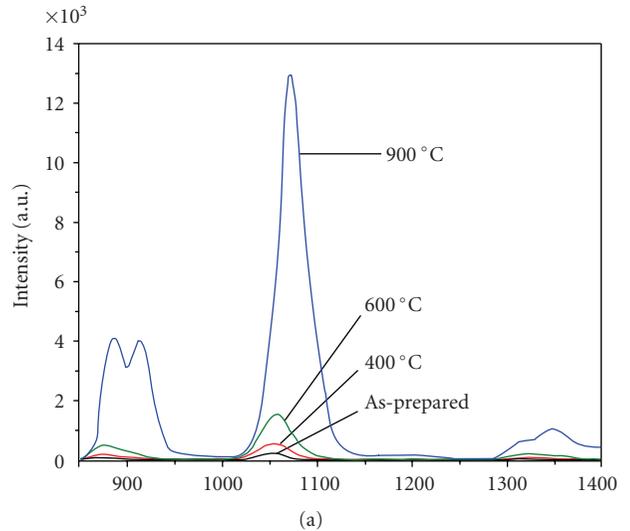


FIGURE 4: Emission spectra of nanoparticles annealed at different temperatures: (a)  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$ , (b)  $\text{LaF}_3:\text{Nd}^{3+}$ .

is just coated on  $\text{LaF}_3:\text{Nd}^{3+}$  core and interaction between the two parts is very weak. When the anneal temperature is  $400^{\circ}\text{C}$ , most solvent remained on nanoparticles is removed. Thus its lifetime is of  $159\ \mu\text{s}$ . When the anneal temperature was raised to  $900^{\circ}\text{C}$ , the interaction between  $\text{SiO}_2$  shell and  $\text{LaF}_3:\text{Nd}^{3+}$  core increases. Since the phonon energy of  $\text{SiO}_2$  ( $1100\ \text{cm}^{-1}$ ) is higher than that of  $\text{LaF}_3$  ( $350\ \text{cm}^{-1}$ ) and the nanoparticles have a larger surface-to-core ratio [16], lifetime of  $\text{Nd}^{3+}$  near or on the surfaces of nanoparticles is slightly shorter than that of  $\text{Nd}^{3+}$  within the core structures and the measured lifetime of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles is decreased.

## 4. Conclusions

To summarize, we have shown that, for the  $\text{SiO}_2$ -coated  $\text{LaF}_3:\text{Nd}^{3+}$  nanoparticles, the  $\text{SiO}_2$  shell has an interesting influence on their  $\text{LaF}_3$  core. When the anneal temperature is  $900^{\circ}\text{C}$ , the change of environment of  $\text{LaF}_3:\text{Nd}^{3+}$  core caused by  $\text{SiO}_2$  shell has become obvious. As a result, Stark splittings of  ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{9/2}$  and  ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{13/2}$  transition peaks and the redshift of  ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{11/2}$  transition peak were observed in their emission spectrum. The fluorescent intensity of  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$  core/shell nanoparticles also has a great improvement, which can be very beneficial for applications.

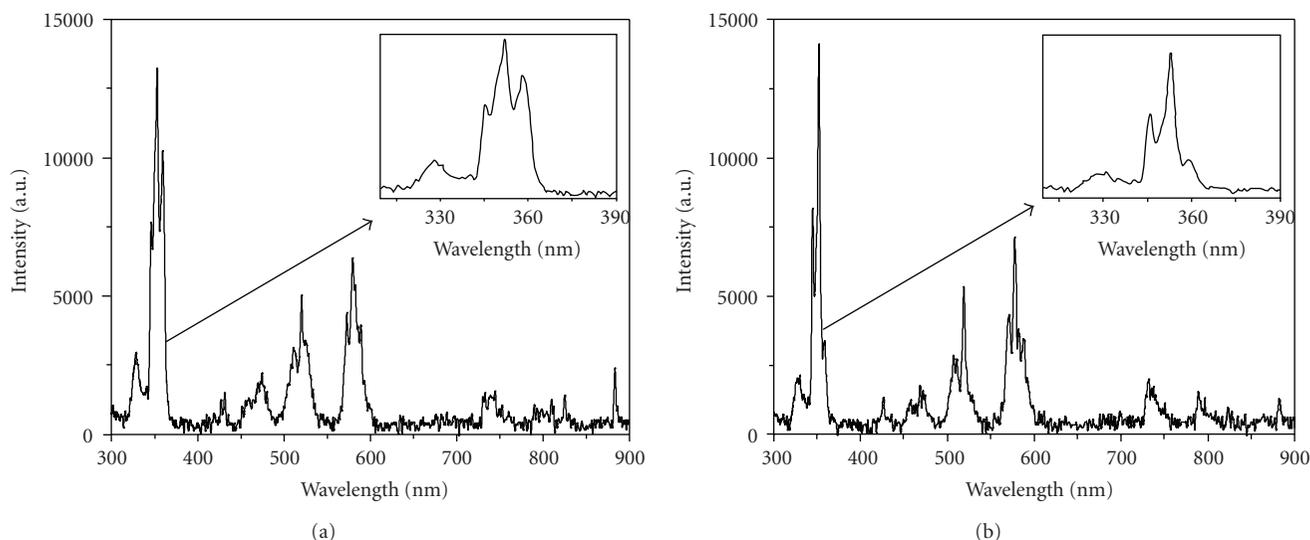


FIGURE 5: Excitation spectra of nanoparticles annealed at 900 °C: (a)  $\text{LaF}_3:\text{Nd}^{3+}/\text{SiO}_2$ , (b)  $\text{LaF}_3:\text{Nd}^{3+}$ .

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