OPTICAL PROPERTIES OF Eu\(^{3+}\) IONS IN LaF\(_3\) NANOCRYSTALS

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ABSTRACT

LaF\(_3\) nanocrystals doped with different concentrations of Eu\(^{3+}\) ions were synthesized by hydrothermal method. The XRD, luminescence and excitation spectra have been studied at room temperature. The ratio of the \(5D_0 \rightarrow 7F_2\) electric dipole transition intensity to the \(5D_0 \rightarrow 7F_1\) magnetic dipole transition intensity (\(R\)) was used to evaluate the asymmetry of Eu\(^{3+}\) site. Judd-Ofelt parameters were calculated from emission spectra and were used to predict the radiative properties of \(5D_0 \rightarrow 7F_1\) transitions also the lifetime of \(5D_0\) level. The small values of \(\Omega_2\) parameter and \(R\) ratio relate to high degree of symmetry of ligand around Eu\(^{3+}\) site in this material.

Keywords: LaF\(_3\):Eu\(^{3+}\) nanocrystals, luminescence, Judd-Ofelt analysis.

1. INTRODUCTION

Rare earth (RE) doped crystals have attracted the attention of scientists due to their wide applications in many optical devices like lasers, light converters, sensors, high-density memories and optical amplifiers [1 - 5]. Compared to the oxide crystals, the fluoride crystals show more featured advantages such as long lifetime, weak nephelauxetic effect and low phonon energy [6 - 8], so these crystals are promising candidates for producing the laser and upconversion devices [4 - 6, 8]. Lanthanum fluoride nanocrystals (LaF\(_3\)) are known with the maximal phonon mode frequency about 380 cm\(^{-1}\) [9], so they are an ideal material for RE dopant because they minimize multiphonon decay processes between energy levels which are very close of the RE\(^{3+}\) ions, this increases the quantum efficiency of the fluorescent transitions. In practice, LaF\(_3\) nanocrystals doped with RE\(^{3+}\) have been used in fibre optics, electrodes, fluorescent lamps, optical amplifiers and radiation applications [9 - 11].

Among the RE\(^{3+}\) ions used to optically activate materials, the Eu\(^{3+}\) ions are mostly chosen due to Eu\(^{3+}\) ions emit narrow-band, almost monochromatic light and have long lifetime of the optically active states. Further, Eu\(^{3+}\) ions have often been used as probes for estimation of local
environment around the Ln$^{3+}$ ions in different matrices [11 - 13]. In this paper, Eu$^{3+}$ ions are used as probe to study the ligand field around RE$^{3+}$ in LaF$_3$ nanocrystals. In addition, optical properties of LaF$_3$:Eu$^{3+}$ are analyzed using Judd–Ofelt theory.

2. EXPERIMENTAL

LaF$_3$ nanocrystals doped with 0; 0.05; 0.1; 0.5 and 1.0 mol% of Eu$^{3+}$ ions were prepared by hydrothermal method. All the chemicals used in our experiment, including lanthanum oxide (La$_2$O$_3$), europium oxide (Eu$_2$O$_3$), ammonium fluoride (NH$_4$F) and glycine (NH$_2$CH$_2$COOH) are of analytic grade without further purification. Annealing process was carried out for 12 hours at temperature of 425 K. The final product was dried in air at 330 K for 12 hours.

The synthesized samples were characterized for their structure by an X-ray diffractometer (SIMENS D5005, Bruker, Germany) with Cu–K$_\alpha$ irradiation (\(\lambda = 1.54056\) Å). The morphology of the samples was observed by using a scanning electron microscope (JEOL-JSM 5410 LV). The optical absorption spectra were recorded in the range of wavelength from 200 to 3000 nm using a spectrometer (Cary-5000). Room temperature photoluminescence (PL) and photoluminescence excitation (PLE) of the samples were measured on a spectrofluorometer (FL 3-22 Jobin Yvon Spex) using 450 W Xe arc lamp as the excitation source.

3. RESULTS AND DISCUSSION

3.1. Structure characterization and morphology

X-ray diffraction patterns of pure LaF$_3$ and LaF$_3$:Eu$^{3+}$ (1 mol% of Eu$^{3+}$ ions) nanocrystals are presented in Fig. 1.

First, quite strong diffraction lines in XRD patterns indicate that LaF$_3$ and LaF$_3$:Eu$^{3+}$ nanoparticles have been crystallized well. Second, all the XRD peaks are unambiguously indexed to hexagonal phase with $P3c1$ space group of LaF$_3$ structure (JCPDS card no. 32-0483 for LaF$_3$) with the following diffraction peaks: (002), (110), (111), (112), (300), (113), (004), (302), (221), (114), (222), (223), (304) and (410). The lattice parameters were calculated to be \(a = 7.174\) Å and \(c = 7.344\) Å.

Next, the obtained results showed that under given experimental conditions no critical change in XRD pattern of Eu$^{3+}$ ion doped (1 mol%) LaF$_3$ was observed, compared to that of pure LaF$_3$, indicating that there was no significant interfacial interaction between Eu and LaF$_3$. Thus, the fact that crystal structure of Eu doped LaF$_3$ was consistent with LaF$_3$ for the investigated concentration range of Eu without presence of Eu own phase or shift in $d$-spacing, this allows to conclude that metallic Eu ions are homogeneously distributed in LaF$_3$ matrix.
The crystallite sizes of LaF$_3$ and LaF$_3$:Eu$^{3+}$ were estimated by using Scherrer’s formula [4] depending on selected peaks. Average sizes of LaF$_3$ and LaF$_3$:Eu$^{3+}$ nanocrystals are about 23 and 25 nm, respectively.

Figure 2 shows FE-SEM image at efficiently high magnification which can provide a rough evaluation about particle size and morphology. From FE-SEM images it can be seen that the average diameter of the particles is approximately 50 nm that is larger than value calculated from Scherrer’s formula. This perhaps is because the crystallites have aggregated, forming bigger particles.

### 3.2. Photoluminescence excitation spectrum and sideband phonon energy

The excitation spectrum of LaF$_3$:Eu$^{3+}$ (1.0 mol %) nanocrystal was recorded in the spectral region 330-560 nm by monitoring the emission at 617 nm ($^5D_{0}→^7F_{2}$ transition) and shown in Fig. 3.

The excitation spectrum consists of the sharp bands due to the f-f transitions from $^6F_{0,1,2}$ of ions Eu$^{3+}$ to the excited levels. The most intense excited band at wavelength of 397 nm corresponds to the $^7F_{0}→^1L_6$ transition, which is often used in fluorescence excitation for Eu$^{3+}$.

The shoulder appears at wavelength around 458 nm can be related to the phonon sideband (PSB), which is used to understand the vibrational modes around the Eu$^{3+}$ [13, 14]. The PSB of Eu$^{3+}$ in LaF$_3$ is associated with the $^7F_{0}→^1D_2$ transition and shown in inset of Fig. 3, in which the $^7F_{0}→^1D_2$ excited transition is the pure electronic transition (PET). The PET is set as zero energy shift, the sideband phonon energy in LaF$_3$ can be calculated to be 290 cm$^{-1}$. This phonon energy is related to the $E_g$ vibration group in LaF$_3$ [9]. The electron phonon coupling constant ($g$) have been calculated by [13]:

$$g = \frac{\int I_{PSB}(\lambda)d\lambda}{\int I_{PET}(\lambda)d\lambda}$$

where $I_{PSB}$ is the intensity of the phonon sideband and $I_{PET}$ is the intensity of the pure electric transition. In LaF$_3$:Eu$^{3+}$ (1.0 mol%), the $g$ value is found to be 0.059. This value is equal to the corresponding values in lead fluoroborate (LFB) glasses [13], but is much lower than that in borotellurite glasses [14].
This behavior shows that the electron-phonon coupling in LaF₃ crystal and LFB glass is weaker than that in borotellurite glasses. Thus, the ionic of Eu³⁺-F⁻ bond in LaF₃ nanocrystal is higher than that Eu³⁺-O²⁻ bond in borotellurite glasses [13].

3.3. Emission spectra

Figure 4 illustrates the emission spectra of LaF₃:Eu³⁺ nanocrystals using the 397 nm excitation wavelength of xenon lamp source. The luminescence lines are interpreted according to Carnall’s paper [15]. Owing to very low phonon energy, the multiphonon relaxation rates from ⁵D₂ and ⁵D₁ levels to ⁵D₀ level are very small, so luminescence from higher excited states of ⁵D₂ and ⁵D₁ were also observed. The emission spectra consist of 12 observed emission bands in Vis and a band in NIR region. There are nine quite strong emission peaks at 488, 509, 525, 534, 553, 582, 590, 617 and 681 nm, which are attributed to ⁵D₂→⁷F₂, ⁵D₂→⁷F₃, ⁵D₁→⁷F₀, ⁵D₁→⁷F₁, ⁵D₁→⁷F₂, ⁵D₁→⁷F₃, ⁵D₀→⁷F₂, ⁵D₀→⁷F₄ transitions, respectively, and three weak emission peaks at 648, 749 and 815 nm corresponding to ⁵D₀→⁷F₁, ⁵D₀→⁷F₃ and ⁵D₀→⁷F₅ transitions, respectively. The ⁵D₀→⁷F₀, S₃, S₅ transitions are forbidden under selection rules, so their intensity are often very weak, however in our case the ⁵D₀→⁷F₀ transition is quite strong. The ⁵D₀→⁷F₂ transition is induced electric dipole (ED), so its intensity depends strongly on the local symmetry around Eu³⁺ ion, polarizability of ligand and energy separation between initial level and final level. The ⁵D₀→⁷F₁ transition is the most intense of all, this is allowed magnetic dipole (MD), the intensity of this transition depends on the host but it is independent local symmetry and reduced matrix elements. So this transition is often used as an internal standard to evaluate the asymmetry of ligand and fluorescent efficiency of ⁵D₀→⁷F₂ transition.

The fluorescence intensity ratio (R) of ⁵D₀→⁷F₂ to ⁵D₀→⁷F₁ transitions of Eu³⁺ ions allows one to estimate the deviation from the site symmetries of Eu³⁺ ions. For LaF₃ doped with 0.05, 0.1, 0.5 and 1.0 mol % of Eu³⁺ ions, the R values are equivalent as shown in table 1. The values of R are less than unity, i.e. the fluorescent efficiency of ⁵D₀→⁷F₁ transition is lower than those of ⁵D₀→⁷F₁ transition. Moreover, these values are smaller than those of many different hosts [13-15]. The lower R values are attributed to the lower asymmetry and covalency around the Eu³⁺ ions in LaF₃ than other hosts. In LaF₃ crystal, Eu³⁺ ion is positioned in a La³⁺ site with C₂ point group symmetry [9], with this symmetry the ⁵D₀→⁷F₀, ⁵D₀→⁷F₁2 transitions split into 1, 3 and 5 components [12], respectively, like in Fig. 4. In principle, the electric current magnetic dipoles are allowed in C₂ symmetry, so both the ⁵D₀→⁷F₁ and ⁵D₀→⁷F₂ transitions have strong intensity [9]. However, the ⁵D₀→⁷F₂ transition is only very intense in the case of highly polarizable, whereas fluoride ligands have low polarizability. This also is the reason for the reduction of R values in the fluoride compounds. Moreover, the luminescence intensity of the ⁵D₀→⁷F₁ transition is stronger than that of ⁵D₀→⁷F₂ transition, this suggests that Eu³⁺ ions take a site with inversion symmetry in LaF₃ crystal [13].

![Figure 4](https://example.com/figure4.png)
3.4. Judd-Ofelt analysis

The Judd-Ofelt (JO) theory was shown to be useful to characterize radiative transitions for RE$^{3+}$-doped solids, as well as aqueous solutions, and to estimate the intensities of the transitions for RE$^{3+}$ ions. This theory defines a set of three intensity parameters, $\Omega_\lambda (\lambda = 2, 4, 6)$, that are sensitive to the environment of the rare-earth ions. Commonly, JO intensity parameters are usually derived from absorption spectrum. However, owing to the special energy level structure of Eu$^{3+}$ ion, these JO parameters could be estimated from the emission spectra. Four main emission peaks $^5D_0 \rightarrow ^7F_{1,2,3,4}$ are used to calculate JO parameters. The $^5D_0 \rightarrow ^7F_1$ is a MD transition and its spontaneous emission probability $A_{md}$ is given by [2,8,9]:

$$ A_{md} = \frac{64\pi^4 \nu^3 n^3 S_{md}}{3h(2J + 1)} \quad (2) $$

where $h$ is the Planck constant, $\nu$ is the wave number of the transition in interest, $J$ is the total angular momentum of the excited state, and $n$ is the refractive index. $S_{md}$ is the MD line strength, which is a constant and independent from the host material. The value of $A_{md}$ can be estimated using the reference value of $A'_md$ published somewhere, and using the relationship $A_{md} = (n/n')^3 A'_md$ [2], where $A'_md$ and $n'$ are spontaneous emission probability and refractive index of the reference material, respectively.

The $^5D_0 \rightarrow ^7F_{2,4,6}$ transitions are an ED partially allowed. The spontaneous emission probabilities $A_{ed}$ of ED transition is given using the following expression:

$$ A_{ed} = \frac{64\pi^4 \nu J^3}{3h(2J + 1)} \sum_{\lambda=2,4,6} \Omega_\lambda \left\| J^{(2)} \right\|^2 \quad (3) $$

where $\nu$ is the wave number of transition $^5D_0 \rightarrow ^7F_\lambda$, $e$ is the electron charge, $\left\| J^{(2)} \right\|^2$ are the squared doubly reduced matrix elements of the unit tensor operator of the rank $\lambda = 2, 4, 6$, and are calculated from intermediate coupling approximation for a transition $|\psi J \rangle \rightarrow |\psi' J' \rangle$. These reduced matrix elements did not nearly depend on host matrix as noticed from earlier studies. Thus the $\Omega_\lambda$ parameters could be evaluated simply by the ratio of the intensity of the $^5D_0 \rightarrow ^7F_{1,2,4,6}$ transitions to the intensity of $^5D_0 \rightarrow ^7F_1$ transition as follows:

$$ \frac{I_J d\nu}{I_J d\nu} = \frac{A(5D_0 \rightarrow 7F_{2,4,6})}{A(5D_0 \rightarrow 7F_1)} = \frac{e^2}{S_{md} \nu J} \left[ \frac{J^{(2)}}{\nu J} \right]^3 \sum_{\lambda=2,4,6} \Omega_\lambda \left\| J^{(2)} \right\|^2 \quad (4) $$

For $^5D_0 \rightarrow ^7F_2$ transition, $U^{(2)} = 0.033; U^{(4)} = U^{(6)} = 0$, $^5D_0 \rightarrow ^7F_4$ transition, $U^{(2)} = 0; U^{(4)} = 0.023; U^{(6)} = 0$ and $^5D_0 \rightarrow ^7F_6$ transition, $U^{(2)} = U^{(4)}, U^{(6)} = 0.003$. Using equation (4) and the reduced matrix elements, the JO parameters were calculated for different concentrations. The results are shown in Table 1 in comparison with different matrix.

The $\Omega_\lambda$ parameters are important to study the symmetry of local structure around RE$^{3+}$ ions and nature of RE–X (X = F, O) bonding. The $\Omega_2$ and $\Omega_6$ are related to the bulk properties such as viscosity and rigidity whereas the $\Omega_4$ is more sensitive to the local environment of the RE$^{3+}$ ions and is often related with the asymmetry of the local crystal field. The $\Omega_2$ and $\Omega_6$ parameters in LaF$_3$ nanocrystal are smaller than those of other hosts. The smaller value of $\Omega_2$ can be attributed to higher symmetry of the ligand field and lower covalency in Eu$^{3+}$-F bond than other hosts.
Optical properties of Eu\(^{3+}\) ions in LaF\(_3\) nanocrystals

whereas the smaller of \(\Omega_6\) parameter shows that the rigidity of the media in which rare earth ions is put into is higher in other hosts. In addition, with the low concentration of Eu\(^{3+}\) in LaF\(_3\) nanocrystal, the values of \(\Omega_2\) and \(R\) are almost independent of the concentration of Eu\(^{3+}\). That is, small changes in the concentration do not alter the asymmetry of the ligand as well as covalency in Eu\(^{3+}\)-F bond.

\[ \Omega_\lambda (x10^{-20} \text{cm}^2) \] JO parameters for Eu\(^{3+}\) doped various hosts.

<table>
<thead>
<tr>
<th>Host matrix</th>
<th>(R)</th>
<th>(\Omega_2)</th>
<th>(\Omega_4)</th>
<th>(\Omega_6)</th>
<th>Refs</th>
</tr>
</thead>
<tbody>
<tr>
<td>LaF(_3) (0.05 mol%)</td>
<td>0.68</td>
<td>1.08</td>
<td>0.73</td>
<td>0.66</td>
<td>Present</td>
</tr>
<tr>
<td>LaF(_3) (0.10 mol%)</td>
<td>0.67</td>
<td>1.06</td>
<td>0.72</td>
<td>0.62</td>
<td>Present</td>
</tr>
<tr>
<td>LaF(_3) (0.50 mol%)</td>
<td>0.68</td>
<td>1.07</td>
<td>0.70</td>
<td>0.60</td>
<td>Present</td>
</tr>
<tr>
<td>LaF(_3) (1.0 mol%)</td>
<td>0.69</td>
<td>1.07</td>
<td>0.68</td>
<td>0.57</td>
<td>Present</td>
</tr>
<tr>
<td>K(<em>2)YF(</em>{10}) (crystal)</td>
<td>-</td>
<td>1.22</td>
<td>1.26</td>
<td>8.55</td>
<td>[17]</td>
</tr>
<tr>
<td>YAlO(_3) (crystal)</td>
<td>-</td>
<td>2.66</td>
<td>6.33</td>
<td>0.80</td>
<td>[17]</td>
</tr>
<tr>
<td>PbFBE (glass)</td>
<td>2.09</td>
<td>2.55</td>
<td>0.36</td>
<td>0</td>
<td>[13]</td>
</tr>
<tr>
<td>L4BE (glass)</td>
<td>3.69</td>
<td>6.34</td>
<td>4.97</td>
<td>5.10</td>
<td>[16]</td>
</tr>
<tr>
<td>B0TN (glass)</td>
<td>2.86</td>
<td>3.29</td>
<td>0.21</td>
<td>0</td>
<td>[14]</td>
</tr>
<tr>
<td>B4TN (glass)</td>
<td>2.28</td>
<td>3.34</td>
<td>0.28</td>
<td>0</td>
<td>[14]</td>
</tr>
</tbody>
</table>

3.5. Radiative properties

The JO parameters have been used to estimate the radiative properties such as the radiative transition rates (\(A_R\), s\(^{-1}\)), branching ratios (\(\beta_{cal}\), %) and stimulated emission cross-section (\(\sigma(\lambda_p)\), \(10^{-22} \text{cm}^2\)) for \(^5\)D\(_0\)\(\rightarrow\)\(^7\)F\(_J\) transitions and radiative lifetime (\(\tau_R\)) of \(^5\)D\(_0\) level of Eu\(^{3+}\) in LaF\(_3\) by using Eqs. in Ref. [18]. In addition, the gain band width (\(\sigma(\lambda_p)\times\Delta\lambda_{eff}, 10^{-28} \text{cm}^3\)) and optical gain (\(\sigma(\lambda_p)\times\tau_{cal}, 10^{-25} \text{cm}^2\text{s}^{-1}\)) are also calculated for \(^5\)D\(_0\)\(\rightarrow\)\(^7\)F\(_J\) transitions. The results are presented in Table 2.

Table 2. The radiative properties of Eu\(^{3+}\) ions doped (1.0 mol \%) in LaF\(_3\).

<table>
<thead>
<tr>
<th>(^5)D(_0)(\rightarrow)</th>
<th>(^7)F(_0)</th>
<th>(^7)F(_1)</th>
<th>(^7)F(_2)</th>
<th>(^7)F(_3)</th>
<th>(^7)F(_4)</th>
<th>(^7)F(_5)</th>
<th>(^7)F(_6)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(A_R)</td>
<td>0</td>
<td>51</td>
<td>40</td>
<td>0</td>
<td>13.2</td>
<td>0</td>
<td>8.6</td>
</tr>
<tr>
<td>(\beta_{cal})</td>
<td>0</td>
<td>45.5</td>
<td>35.4</td>
<td>0</td>
<td>11.7</td>
<td>0</td>
<td>7.7</td>
</tr>
<tr>
<td>(\beta_{mes})</td>
<td>6.6</td>
<td>48.3</td>
<td>32</td>
<td>0.98</td>
<td>10.6</td>
<td>0.9</td>
<td>0.6</td>
</tr>
<tr>
<td>(\sigma(\lambda_p))</td>
<td>0</td>
<td>5.6</td>
<td>4.2</td>
<td>0</td>
<td>2.0</td>
<td>0</td>
<td>1.6</td>
</tr>
<tr>
<td>(\sigma(\lambda_p)\times\Delta\lambda_{eff})</td>
<td>0</td>
<td>3.2</td>
<td>2.9</td>
<td>0</td>
<td>1.5</td>
<td>0</td>
<td>1.9</td>
</tr>
<tr>
<td>(\sigma(\lambda_p)\times\tau_{cal})</td>
<td>0</td>
<td>49.7</td>
<td>35.5</td>
<td>0</td>
<td>17.7</td>
<td>0</td>
<td>14.2</td>
</tr>
</tbody>
</table>

The predicted branching ratio (\(\beta_{cal}\)) of \(^5\)D\(_0\)\(\rightarrow\)\(^7\)F\(_1\) transition get a maximum value and is 45.2 % whereas the measured ratio (\(\beta_{mes}\)) is 48.3 %, thus there is a good agreement between experimental and calculated branching ratios. The branching ratio, gain band width and optical gain of this transition are larger than those of other transitions. These results suggest that the \(^5\)D\(_0\)\(\rightarrow\)\(^7\)F\(_1\) transition of Eu\(^{3+}\) ions in LaF\(_3\) nanocrystal is found to be suitable for developing the fibre optics [19].

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4. CONCLUSIONS

LaF$_3$:Eu$^{3+}$ nanocrystals were prepared by hydrothermal method. The XRD patterns indicate that LaF$_3$ nanoparticles have been crystallized well. From the excitation spectrum, the PSB was found with the energy phonon about 290 nm, which related to the $E_g$ vibration group in LaF$_3$. The smaller value of $g$ parameter than those in borotellurite glasses shows that the electron phonon coupling in LaF$_3$:Eu$^{3+}$ nanocrystal is weaker than that of borotellurite glasses.

The optical properties of Eu$^{3+}$-doped LaF$_3$ nanocrystals have been investigated. The small value of $R$ and $\Omega_2$ parameter shows that the coordination structure surrounding the Eu$^{3+}$ ions has high symmetry and Eu$^{3+}$-F bond has low polarizability. The radiative parameters show that the $^5D_{3/2} \rightarrow ^5F_1$ transition of Eu$^{3+}$ ions in LaF$_3$ nanocrystals is very useful fiber optic amplifier.

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TÓM TÁT

TÍNH CHẤT QUANG CỦA CÁC ION Eu$^{3+}$ TRONG TINH THỂ NANO LaF$_3$

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Các hạt nano LaF$_3$ phát tia ion Eu$^{3+}$ với các nóng độ khác nhau được chế tạo bằng phương pháp thủy nhiệt. Các phép đo XRD, huỳnh quang và kích thích huỳnh quang được thực hiện tại nhiệt độ phòng. Từ dữ liệu cure người đã huỳnh quang $^5$D$_0$$\rightarrow$$^7$F$_2$ (dipole dien) và $^5$D$_0$$\rightarrow$$^7$F$_1$ (dipole từ) (R) được sử dụng để đánh giá tính bất đối xứng của trường ligand xung quanh ion Eu$^{3+}$. Các thông số Judd – Ofelt được tính toán từ phổ huỳnh quang đã được sử dụng để dự đoán các tính chất phát xạ của các chuyển đổi $^5$D$_0$$\rightarrow$$^7$F$_2$ cũng như thời gian sống của mức $^5$D$_0$. Giá trị nhỏ của thông số $\Omega_2$ và tỉ số $R$ liên quan đến tính đối xứng cao của trường ligand xung quanh ion Eu$^{3+}$ trong vật liệu này.

Từ khóa: tính thể nano LaF$_3$:Eu$^{3+}$, huỳnh quang, phân tích Judd-Ofelt.