Photoluminescence and cathodoluminescence properties of Li+ doped Gd$_{1.88}$Eu$_{0.12}$O$_3$

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Li$^+$-doped cubic Gd$_{1.88}$Eu$_{0.12}$O$_3$ phosphors were synthesized at 1200°C in air by co-precipitation (CP) and solid state reaction (SS) methods. X-ray diffraction analysis revealed that, regardless of synthetic method, the average crystallite size for Li$^+$-free Gd$_{1.88}$Eu$_{0.12}$O$_3$ was approximately 400 nm, then increased with the amount of Li$^+$ and at 20 mol %, reached about 2.3 μm. Under excitation of the charge transfer band of Eu$^{3+}$ at 245 nm, Gd$_{1.88}$Eu$_{0.12}$O$_3$ exhibited a dominant photoluminescence (PL) red emission peak at 611 nm assigned to the electric dipole transition $^5D_{0} \rightarrow ^7F_2$ of Eu$^{3+}$. The red emission peak intensity increased consistently with the amount of Li$^+$-doping, while CP method was found to be effective in improving the red emission intensity at lower amounts of Li$^+$-doping from 0 to 8 mol %. Cathodoluminescence (CL) property was studied by mounting the synthesized phosphor on a vacuum fluorescent display (VFD) operated at an anode voltage of 50 V. The Gd$_{1.88}$Eu$_{0.12}$O$_3$ exhibited a similar spectrum with an intense red emission peak at 611 nm, and the highest luminance intensity for the CL red emission was achieved for 8 mol % Li$^+$-doped Gd$_{1.88}$Eu$_{0.12}$O$_3$ synthesized by SS method. The evaluation results of PL and CL properties suggested that, besides the crystallite size of Gd$_{1.88}$Eu$_{0.12}$O$_3$, dispersion property of Eu$^{3+}$ ions in the host Gd$_2$O$_3$ was an important factor for improving luminescent properties of Gd$_{1.88}$Eu$_{0.12}$O$_3$. Moreover, under the present VFD operating condition at the low excitation voltage, it was thought to be essential for improving CL emission intensity to maintain a sufficient surface area of Gd$_{1.88}$Eu$_{0.12}$O$_3$.

Key-words : Gd$_2$O$_3$:Eu$^{3+}$, Li$^+$, Photoluminescence, Cathodoluminescence, Dispersion property, Crystallite size, Specific surface area

1. Introduction

Field Emission Displays (FEDs) have been widely used for televisions, personal computers and cell phones in our daily life to replace the conventional cathode ray tube (CRT) displays.\(^{11}\) In the past decades, most of the interest has been focused on the materials for FEDs operating at low electron beam excitation voltages ($\leq$10 kV).\(^{21}\) However, under the low excitation voltages, there is no ideal material with sufficient brightness, fast response and long term stability used for the modern luminescence devices.\(^{23}\) Former researches have mainly used sulfide red phosphors such as Y$_2$O$_2$S:Eu$^{3+}$, SrGa$_2$S$_4$:Eu$^{3+}$, ZnS:Ag,Cl, (Zn/Cd)S:Cu etc.\(^{47}\) Unfortunately, the sulfide phosphors are unstable under electron bombardment in vacuum, which leads to the chemical degradation of phosphor layers and emit sulfide-containing gases under the operation condition.\(^{3,58}\) On the other hand, due to the sharp lines, high efficiency and stability in high vacuum, rare-earth sesquioxides have received increased attention and their luminescence properties have been investigated.\(^{79-111}\) Europium(III)-doped Gd$_2$O$_3$ phosphor (Gd$_{2-}$Eu$_{0.9}$O$_3$) is one of the attractive red emitting rare-earth sesquioxide phosphors with potential applications in various lighting and display devices.\(^{122}\) Both Gd$_2$O$_3$ and Eu$_2$O$_3$ have three different kinds of cubic, monoclinic and hexagonal phases. It is reported that the smaller electronegativity of Gd$^{3+}$ permits an easier charge transfer from the 2p orbital of O$^2-$ to the 4f orbital of Eu$^{3+}$, and the $^7S_{6,7,9}$ $\rightarrow$$^5D$ transitions of Gd$^{3+}$ overlap in the charge transfer band (CTB), which leads to efficient energy transfer from Gd$^{3+}$ to Eu$^{3+}$ in Gd$_{2-}$Eu$_{0.9}$O$_3$. Therefore, cubic Gd$_{2-}$Eu$_{0.9}$O$_3$ exhibits stronger and red-shift CTB of the charge transfer edge, which allows sharper red emission at 611 nm than the monoclinic Gd$_{2-}$Eu$_{0.9}$O$_3$.\(^{13,14}\)

However, compared with commercial red phosphors like Zn$_2$Cd$_3$S:Ag$^{+}$ and Y$_2$O$_2$S:Eu$^{3+}$,\(^{21}\) the red emission intensity of Gd$_{2-}$Eu$_{0.9}$O$_3$ is not sufficient for practical applications. There are two main approaches to improve the luminescence properties of Gd$_{2-}$Eu$_{0.9}$O$_3$: morphology and/or structure controlling, and with hetero-element ions. Several Gd$_{2-}$Eu$_{0.9}$O$_3$ phosphors like nanotubes,\(^{15}\) nanowires,\(^{16}\) and nanospherical particles\(^{17}\) have been synthesized. However, during synthesis of the nanometer-sized Gd$_{2-}$Eu$_{0.9}$O$_3$, lots of chemical reagents like urea and dispersant are used, which introduces impurity ions and leads to form non-radiation centers. For the second approach, alkali metal ions are useful dopants to enhance the emission intensity because of the flux effect and the formation of oxygen vacancies.\(^{21,39-22}\) Moreover, due to the radius smaller than the Gd$^{3+}$, Li$^+$ is an attractive secondary dopant (compare with the Na$^+$ and K$^+$) for
host Gd₂O₃, which contributes to increase local distortion of the crystal field surrounding the Eu³⁺ activator. In our previous study on Gd₂₋ₓEuₓO₃ with optimized Eu³⁺ concentration of 12 mol% (Gd₁₈ₓEuₓ₁₂O₃), the PL red emission intensity was found to increase approximately three times by Li⁺-doping at 12 to 16 mol%. Moreover, the thermal stability of the cubic Gd₁₈₋ₓEuₓ₁₂O₃ was significantly improved by the Li⁺-doping, and even after annealing for 72 h at 1300°C, which exceeds the cubic-monoclinic phase transformation temperature, the relative fraction of the cubic phase in the 12 mol% Li⁺-doped Gd₁₈₋ₓEuₓ₁₂O₃ was measured to be 90%. As a part of our study on luminescent properties, here we report a comparative study of PL and cathodoluminescence (CL) properties of Li⁺-doped cubic Gd₁₈₋ₓEuₓ₁₂O₃. In the present work, Gd₂O₃–Eu₂O₃ mixed powder was prepared by using co-precipitation technique and cubic Gd₁₈₋ₓEuₓ₁₂O₃ phosphors with different amounts of Li⁺ were synthesized by heat treatment at 1200°C in air for 3 h. PL properties were evaluated by using a conventional spectrophotometer, while CL emission properties under low excitation voltage were investigated by mounting the synthesized Gd₁₈₋ₓEuₓ₁₂O₃ phosphor on a vacuum fluorescent display (VFD). The resulting PL and CL properties were discussed by comparison with those synthesized by the conventional solid solution method.

2. Experimental procedures

2.1 Synthesis of Li⁺-doped cubic Gd₁₈₋ₓEuₓ₁₂O₃ phosphors

Gadolinium oxide (Gd₂O₃), europium oxide (Eu₂O₃) and lithium carbonate (Li₂CO₃) were used as starting powders. All the powders were purchased from Mitsuwa Chemical Company, Japan (99.99% purity). Appropriate amounts of Gd₂O₃ and Eu₂O₃ were completely dissolved in a HNO₃ aqueous solution with pH = 1. Through the whole process, the aqueous solution was stirred by a magnetic stirrer. The precipitation was carried out by adding aqueous NH₄OH solution until the solution reached a pH of 8. The precipitate was separated by filtration and dried at 100°C for 3 h. The dried powder was calcined at 1200°C in air for 3 h. In a typical synthesis, a proper amount of the calcined powder was mixed with Li₂CO₃ as a source of Li⁺, and after annealing for 72 h at 1300°C, which exceeds the cubic-monoclinic phase transformation temperature, the relative fraction of the cubic phase in the 12 mol% Li⁺-doped Gd₁₈₋ₓEuₓ₁₂O₃ was measured to be 90%.

2.2 Characterizations

Room temperature X-ray diffraction (XRD; X’pert Pro α1, Philips) analysis was performed on the 1200°C heat-treated samples using CuKα radiation (λ = 0.15405 nm). The average crystallite size of Gd₁₈₋ₓEuₓ₁₂O₃ phosphors was evaluated by the Scherrer equation:

\[ D = \frac{0.9λ}{B\cos θ} \]

where D is the average crystallite size, λ is the X-ray wavelength (λ = 0.15405 nm), B is the full width at half maximum of the observed peak, and the factor of 0.9 for spherical objects was used in this study.

Morphology and microstructure of Li⁺-doped Gd₁₈₋ₓEuₓ₁₂O₃ phosphors was observed using a scanning electron microscope (SEM, JSN-6360LV , JEOL) at 25 kV acceleration voltages.

PL excitation and emission spectra were record by a spectrophotometer (F-7000, Hitachi) with a 150 W Xenon lamp as the excitation source. CL emission properties of the synthesized Li⁺-doped Gd₁₈₋ₓEuₓ₁₂O₃ phosphors were studied by mounting the phosphor on a VFD operated at an anode voltage of 50 VDC (Pulse Width of 100 msec, and Pulse Duty of 1/60 generated by FC110, YOKOGAWA, Japan) with an average current density of 1.3 mA/cm². The CL emission spectrum and the luminance intensity at room temperature were recorded and measured, respectively by a spectroradiometer (TOPCON SR-3A, Topcon Technohouse Corporation, Japan). This VFD mount test was performed at Noritake Itron Corporation, Japan.

3. Result and discussion

3.1 Crystalline phase and morphology

Powder XRD patterns for 1200°C-synthesized Gd₁₈₋ₓEuₓ₁₂O₃ synthesized by co-precipitation (CP) and solid solution (SS) methods are shown in Fig. 1. The diffraction peaks detected for each sample were identical to those for the standard powder diffraction file for cubic Gd₂O₃ (JCPDS No. 86-2477, space group: Ia-3). Even for the samples with 20 mol% Li⁺, no

![Fig. 1. XRD patterns for Gd₁₈₋ₓEuₓ₁₂O₃ doped with different amounts of Li⁺ synthesized by CP and SS methods after heat treatment at 1200°C in air for 3 h ( indicates cubic Gd₂O₃).](image)
additional crystalline phases containing Li⁺ species were detected.

Figure 2 shows the average crystallite size for Gd₁.₈₈Eu₀.₁₂O₃ doped with different amounts of Li⁺ synthesized by CP and SS methods after heat treatment at 1200°C in air for 3 h.

The samples synthesized by CP method also showed the same tendency. However, at the Li⁺ doping from 0 to 8 mol %, the SSA for Gd₁.₈₈Eu₀.₁₂O₃ synthesized by CP method was apparently lower than that by SS method. Above 8 mol %, SSA values were lower than 0.4 m²/g and compatible with those synthesized by SS method.

3.2 PL properties

Figure 5 demonstrates PL excitation spectrum ($\lambda_{ex} = 611$ nm) and emission spectrum ($\lambda_{em} = 245$ nm) for 12 mol % Li⁺-doped Gd₁.₈₈Eu₀.₁₂O₃ synthesized by CP method. The spectra were well consistent with those synthesized by SS method in our previous study and the excitation spectrum exhibits three main peaks at 245, 255 and 277 nm. The highest peak at 245 nm is attributed to the charge transfer band (CTB) between O²⁻ and Eu³⁺.¹³,¹⁴ The peaks at 255 and 277 nm superimposed on the CTB of Eu³⁺ are due to the Gd₂O₃ host excitation, and are assigned to the $8S = 6D$ and $8S = 6I$ transitions of Gd³⁺, respectively. The weak peak at 311 nm can be assigned to the $8S = 6P$ transition of Gd³⁺. The samples synthesized by CP method also showed the same tendency. However, at the Li⁺ doping from 0 to 8 mol %, the SSA for Gd₁.₈₈Eu₀.₁₂O₃ synthesized by CP method was apparently lower than that by SS method. Above 8 mol %, SSA values were lower than 0.4 m²/g and compatible with those synthesized by SS method.
can be assigned to the $^5D_0 \rightarrow ^7F_0$ transition and the emission peak at 591 nm is attributed to the $^5D_0 \rightarrow ^7F_1$ magnetic dipole transition. The most intense emission peak at 611 nm and another weak peak at 628 nm are due to the $^5D_0 \rightarrow ^7F_2$ electric dipole transition.

Typical PL emission spectra ($\lambda_{ex} = 245$ nm) for the Gd$_{1.88}$Eu$_{0.12}$O$_3$ phosphors synthesized in this study are shown in Fig. 6. There was a tendency for the dominant red emission peak intensity at 611 nm to increase consistently with the amount of Li$^+$-doping. Then, the values of the peak intensity at 611 nm for the Gd$_{1.88}$Eu$_{0.12}$O$_3$ phosphors were plotted and analyzed. As shown in Fig. 7(a), the intensity for the phosphor synthesized by SS method increased with increasing amount of Li$^+$-doping and reached a maximum at 20 mol% Li$^+$-doping. On the other hand, the intensity for the Li$^+$-free Gd$_{1.88}$Eu$_{0.12}$O$_3$ synthesized by CP method was high and compatible with that achieved for the 16 mol% Li$^+$-doped Gd$_{1.88}$Eu$_{0.12}$O$_3$ synthesized by SS method. Then, the intensity increased slightly to reach a maximum at 8 to 20 mol%, and was close to that for the 20 mol% Li$^+$-doped Gd$_{1.88}$Eu$_{0.12}$O$_3$ synthesized by SS method.

The dominant emission peak at 611 nm due to the $^5D_0 \rightarrow ^7F_2$ electric dipole transition is hypersensitive to the symmetry of local environment around Eu$^{3+}$ ion, while that due to the $^5D_0 \rightarrow ^7F_1$ transition at 591 nm is attributed to a magnetic dipole transition, which is not affected by the inverse site symmetry. It is well known that the relative intensities of the $^5D_0 \rightarrow ^7F_1$ and $^5D_0 \rightarrow ^7F_2$ emissions strongly depend on the local symmetry of Eu$^{3+}$, and the $^5D_0 \rightarrow ^7F_2/^5D_0 \rightarrow ^7F_1$ emission intensity ratio (asymmetry ratio) indicates the degree of distortion from the inversion symmetry of the local environment around Eu$^{3+}$ ion in the host lattice. In our previous study on Li$^+$-doped Gd$_{1.88}$Eu$_{0.12}$O$_3$ synthesized by SS method, it was experimentally demonstrated that the dominant PL red emission peak intensity at 611 nm was proportional to the asymmetric ratio, and the asymmetric ratio increased consistently with the amount of Li$^+$-doping. Then, the asymmetric ratios were evaluated for the phosphors synthesized by CP method. The values were plotted as a function of amount of Li$^+$-doping and compared with those synthesized by SS method. As shown in Fig. 7(b), the asymmetric ratio increased with the amount of Li$^+$-doping, however at...
all the amount of Li\textsuperscript{+}-doping in the range of 0 to 20 mol\%%, the asymmetric ratio for Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} synthesized by CP method was found to be lower than that synthesized by SS method.

Generally, large surface area carries large number of defects into the host lattice. Defects have a serious drawback in PL emission intensity since they provide non-radiative recombination routes for the electrons and holes. If the defect density is sufficiently reduced by crystallite growth, PL emission intensity would be improved.\textsuperscript{20,29-30} In the present study, PL red emission intensity for the Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} synthesized by SS method was successfully enhanced by host Gd\textsubscript{2}O\textsubscript{3} crystallite growth via liquid phase-assist sintering. However, as shown in Figs. 2 and 3, the Li\textsuperscript{+}-free Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} synthesized by CP method exhibited a fine-grained microstructure with an average crystallite size of approximately 400 nm. One possible reason for the high PL emission intensity is due to the improved dispersion property of Eu\textsuperscript{3+} ions doped in the host Gd\textsubscript{2}O\textsubscript{3}. During the synthesis process using co-precipitation technique, Gd\textsubscript{2}O\textsubscript{3} and Eu\textsubscript{2}O\textsubscript{3} were converted to Gd(NO\textsubscript{3})\textsubscript{3} and Eu(NO\textsubscript{3})\textsubscript{3}, respectively. The resulting soluble salts could be well and uniformly mixed at the molecular level, which leading to minimize the energy trapped by the non-radiative recombination center during the energy migration process between one Eu\textsuperscript{3+} ion to another one caused by segregation of Eu\textsuperscript{3+} ions in the host Gd\textsubscript{2}O\textsubscript{3}. As a result, relatively high PL emission intensity was thought to be achieved for the Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} at the Li\textsuperscript{+}-doping from 0 to 8 mol\%.

### 3.3 CL properties

The Li\textsuperscript{+}-doped Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} was mounted on a VFD, and under the VFD operating condition at an anode voltage of 50 V, CL properties were studied. As a typical result, Fig. 8 presents a CL emission spectrum for 8 mol\% Li\textsuperscript{+}-doped Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} synthesized by CP method. Characteristic emission peaks due to the \( f-f \) transition of Eu\textsuperscript{3+} ions are observed. The dominant emission peak is located at 611 nm assigned to the \( D_{0.5}\textrightarrow T_{2.2} \) electric dipole transition of Eu\textsuperscript{3+}. Thus, the Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} exhibits intense CL red emission under the VFD operating condition in this study.

Figure 9(a) shows luminance intensity evaluated for the dominant CL red emission of Li\textsuperscript{+}-doped Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3}. In contrast to the liner relation between the amount of Li\textsuperscript{+} and PL emission intensity shown in Fig. 7(a), Li\textsuperscript{+}-doped Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} synthesized by SS method exhibited a maximum luminance intensity at 8 mol\% Li\textsuperscript{+}, while above this amount, the intensity decreased with the amount of Li\textsuperscript{+}. On the other hand, when Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} was synthesized by CP method, the manimum luminance intensity (at 0 mol\% Li\textsuperscript{+}) remarkably improved to 31.3 cd/m\textsupersq, and the intensity achieved a maximum at 4 mol\% Li\textsuperscript{+}. However, this intensity (37.3 cd/m\textsupersq) was apparently lower than that achieved for the 8 mol\% Li\textsuperscript{+}-doped Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} synthesized by SS method (53.9 cd/m\textsupersq).

Figure 9(b) demonstrates relation between the SSA and the luminance intensity for Gd\textsubscript{1.88}Eu\textsubscript{0.12}O\textsubscript{3} synthesized in this study. Regardless of synthesis method, the luminance intensity reaches a maximum at around 0.8 m\textsupersq/g, while below this value, the intensity decreases with increasing amount of Li\textsuperscript{+}-doping.

At electron beam excitation voltages ranging from 1 to 10 kV, the penetration depth of electrons into the solid is well described by Feldman\textsuperscript{31} using the high-energy excitation model. Based on the model, for example, Liu et al. reported that the electron penetration depth at 5 kV for rare-earth cation-doped SrIn\textsubscript{2}O\textsubscript{4} was estimated as 67.44 nm.\textsuperscript{32} At high excitation voltages, CL properties should be governed by material factors such as crystallinity of host material and dispersion property of activator ions in the host lattice. On the other hand, under our present VFD operating condition at an anode voltage of 50 V, the electron penetration depth must be extremely shallow. Therefore, it is...
essentially required for enhancing the luminance intensity to maintain a sufficient surface area of a phosphor mounted in a VFD. In this study, crystallinity of Gd_{1.88}Eu_{0.12}O_3 and dispersion property of Eu^{3+} in the host lattice could be improved in association with decreasing surface area of Gd_{1.88}Eu_{0.12}O_3. Thus, the SSA value of 0.8 m²/g is considered as an approximate lower limit that can offer possibilities for enhancing luminance intensity.

4. Summary

In this study, Li^{+}-doped Gd_{1.88}Eu_{0.12}O_3 phosphors were synthesized at 1200°C in air by co-precipitation (CP) and solid solution (SS) methods. The PL and CL properties of the synthesized phosphors were studied and discussed based on the material factors in terms of the average crystallite size, dispersion property of Eu^{3+} ions in the host Gd_2O_3 and specific surface area (SSA). The results are summarized as follows:

(1) The average crystallite size for Gd_{1.88}Eu_{0.12}O_3 synthesized by CP method was closed to that synthesized by SS method at all the amount of Li^{+}-doping from 0 to 20 mol %. The average crystallite size at 0 mol % Li^{+} was approximately 400 nm, then increased consistently with amount of Li^{+}-doping. The maximum average crystallite size achieved at 20 mol % Li^{+} was about 2.3 μm.

(2) Under the excitation of charge transfer band between O^{2-} and Eu^{3+} at 245 nm, Gd_{1.88}Eu_{0.12}O_3 exhibited a dominant red emission peak at 611 nm attributed to the {^7}D_0→{^7}F_2 electric dipole transition, and the intensity of the dominant red emission peak increased consistently with the average crystallite size of Gd_{1.88}Eu_{0.12}O_3.

(3) The dispersion property of Eu^{3+} ions doped in the host Gd_2O_3 was thought to be improved by using CP method, which could lead to the relatively high PL red emission intensity achieved for the Gd_{1.88}Eu_{0.12}O_3 at the Li^{+} doping from 0 to 8 mol %.

(4) The Li^{+}-doped cubic Gd_{1.88}Eu_{0.12}O_3 phosphor mounted on a VFD showed room-temperature red CL at an anode voltage of 50 V, and the maximum luminescence intensity for the CL red emission was achieved for 8 mol % Li^{+}-doped Gd_{1.88}Eu_{0.12}O_3 synthesized by SS method.

(5) Under the present VFD operation condition at the low excitation voltage, the penetration depth of electrons into the Gd_{1.88}Eu_{0.12}O_3 was considered to be extremely shallow. Therefore, it could be essential for enhancing the luminescence intensity to maintain a sufficient surface area of the Gd_{1.88}Eu_{0.12}O_3 mounted on a VFD. As a result, the SSA value of approximately 0.8 m²/g was suggested as a lower limit required for improving CL emission intensity.

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