Luminescence Investigation on Ultraviolet-Emitting Rare-Earth-Doped Phosphors Using Synchrotron Radiation

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ABSTRACT: Three series of new ultraviolet-emitting Ca₉Y(PO₄)₇:Ln³⁺ (Ln = Ce, Gd, Pr) phosphors were synthesized, and their luminescence was investigated. Under vacuum ultraviolet excitation Ca₉Y(PO₄)₇:Ce³⁺ phosphors emit UVA light with one broad emission centered at 346 nm, on account of the 5d¹ → 4f⁰ transition of Ce³⁺ ions; the optimal doping concentration of these phosphors is 0.2 mol. Ca₉Y(PO₄)₇:Gd³⁺ phosphors show a strong 4f² → 4f⁰ transition and a sharp UBV emission band at 312 nm; the optimal doping concentration of these phosphors is 0.7 mol. The PL spectra of Ca₉Y(PO₄)₇:Pr³⁺ show two broad UVA emission bands centered between 230 and 340 nm, owing to the 4f⁵5d¹ → 4f⁰ transition of Pr³⁺ ions; the optimal doping concentration of these phosphors is 0.2 mol. Under 172 nm excitation, we found that the luminescence intensity of the UVA-emitting Ca₉Y(PO₄)₇:Pr³⁺:0.2Ce³⁺ is 0.3675 times that of Ba₅Sr₂O₃:0.05Pb²⁺, that of the UBV-emitting Ca₉Y(PO₄)₇:0.7Gd³⁺ is 1.7 times that of Y₂O₃(BO₃)₄:0.25Gd³⁺, and that of the UVC-emitting Ca₉Y(PO₄)₇:0.2Pr³⁺ is 1.5 times that of LaPO₄:0.1Pr³⁺. The thermal stability investigation indicated that the luminescence decay was only 9.2%, 18.2%, and 10.3% for Ca₉Y(PO₄)₇:0.2Ce³⁺, Ca₉Y(PO₄)₇:0.7Gd³⁺, and Ca₉Y(PO₄)₇:0.2Pr³⁺ at 250 °C relative to that at ambient temperature, respectively. The Ca₉Y(PO₄)₇:Ln³⁺ (Ln = Ce, Gd, Pr) phosphors exhibit high emission efficiency and excellent thermal stability.

1. INTRODUCTION

In recent years, ultraviolet radiation (UVR) has been extensively used in a variety of applications such as examination of counterfeit banknotes and insect traps,¹ production of vitamin D₃ and photochemical reactions.² It has also been used in medical applications such as treatment of psoriasis,³ induction of ripening.⁴ UVR can be generally divided into three categories: ultraviolet A (UVA, 400–320 nm), ultraviolet B (UVB, 320–280 nm), and ultraviolet C (UVC, 280–200 nm). More than 98% of UVA radiation reaches the surface of the earth. Most of the UVB radiation is absorbed by the atmosphere, and only ~2% of UVR radiation reaches the surface of the earth. UVC radiation is completely scattered and absorbed by O₃, O₂, and N₂ into the atmospheric layer.⁸

For designing ultraviolet-emitting phosphors, the most suitable activators used are Ce³⁺, Gd³⁺, Pr³⁺, and Pb²⁺ ions. Tian et al.⁹ studied the PLE spectra of GdPO₄:Ce³⁺ and found that five crystal field splitting components were present at 206, 214, 236, 256, and 274 nm; these components were formed because of the crystal field splitting of Ce³⁺ 5d states in GdPO₄:Ce³⁺. Under 155-nm excitation, strong UVA emissions at around 343 nm of Ce³⁺ (5d¹ → 4f⁰) and 311 nm of Gd³⁺ (6P₇/₂ → 8S₇/₂) can be observed; this observation implies that the efficient energy is transferred between the host lattice and Ce³⁺. Narrow-band UVB-emitting phosphors of YAl₅(BO₃)₄:Gd³⁺ and LaPO₄:Gd³⁺, Pr³⁺ have been observed by Yokosawa et al.¹⁰ and Okamoto et al, respectively.¹¹ They reported that the emission spectra of YAl₅(BO₃)₄:Gd³⁺ and LaPO₄:Gd³⁺,Pr³⁺ at 313 and 312 nm, respectively, originated from the 6P₇/₂ to 8S₇/₂ transition of Gd³⁺ ions. The authors reported that the emission intensity of (La₀.₆₅Gd₀.₃₅)₀.₉₅Pr₀.₀₅PO₄ is approximately 1.6 times that of Y₀.₇₅Gd₀.₂₅Al₀.₃₉BO₄ under 172 nm excitation. According to Yuan et al.,¹² NaLaP₂O₇:Pb³⁺ phosphor shows three excitation bands between 160 and 225 nm; these bands are attributed to the 4f⁵5d¹ → 4f⁰ transition of Pr³⁺ ions. The PL spectra show a moderately strong 4f⁵5d¹ → 4f² (6H₅₂ → 6P₃₂; 1G₄ → 1D₂; 3P₃ → 1I₆) transition and broad emission bands. Folkerts et al.¹³ described Pb²⁺-doped alkaline earth sulfates and indicated that the PL spectra of CaSO₄:Pb²⁺ show a broad emission band and an excitation band with a maximum at 235 and 220 nm owing to the sp → s² (2P₁₁ → 1S₀) and sp → sp transitions of Pb²⁺ ions, respectively.

In this study, the luminescence property, thermal stability, and emission efficiency of UV-emitting Ca₉Y(PO₄)₇:Ln³⁺ (Ln = Ce, Gd, Pr) phosphors have been reported. Our results indicated that Ca₉Y(PO₄)₇:Ln³⁺ (Ln = Ce, Gd, Pr) phosphors have good thermal stability and high luminous efficiency and, therefore, may serve as promising ultraviolet-radiation phosphors for application in xenon excimer discharge lamps.

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2. EXPERIMENTAL SECTION

2.1. Materials and Synthesis. Polycrystalline phosphors of Ca9-(Y1−xCe3+x)(PO4)7, Ca9(Y1−xGd3+y)(PO4)7, and Ca9(Y1−xPr3+z)(PO4)7, were prepared using a high-temperature solid-state reaction. Initially, the constituent raw materials CaCO3, Y2O3 (99.99%), (NH4)2HPO4, CeO2 (99.99%), Gd2O3 (99.99%), and Pr2O3 (99.99%) (all analytically pure and high purity from Aldrich Chemicals) were weighed in stoichiometric proportions, mixed thoroughly ground in an agate mortar, and then placed in an alumina crucible, which was heated at 1250 °C for 8 h and then cooled slowly to room temperature. The Ca9(Y1−xCe3+x)(PO4)7 phosphors were then reduced at 1000 °C for 6 h under a reducing atmosphere of 15% H2/85% N2 in an alumina boat.

2.2. Materials Characterization. The phase purity of the as-prepared samples was checked by powder X-ray diffraction (XRD) analysis with a Bruker AXS D8 advanced automatic diffractometer with Cu Kα radiation (λ = 1.5418 Å) over the angular range 10° ≤ 2θ ≤ 80°, operating at 40 mA. The photoluminescence (PL) and photoluminescence excitation (PLE) spectra were recorded upon excitation with vacuum ultraviolet (VUV) radiation, which was dispersed from a high-flux beamline with a 6 m cylindrical grating monochromator (CGM) coupled to the 1.5 GeV storage ring at the National Synchrotron Radiation Research Center (NSRRC) in Taiwan. This CGM beamline spans the energy range 4–40 eV with a resolving power of 40,000; the highest photon flux is about 2 × 1012 photons/s. The incident VUV light was conducted through a gold mesh with about 90% transmission; the excitation light further transmitted a CaF2 window before being irradiated onto the sample. To remove the high-order light, the excitation light further transmitted a CaF2 window before being irradiated onto the sample. Emission of the PL was detected by an Aladdin Photoluminescence Spectrophotometer Model Fluoromax-3 spectrophotometer. The microstructure of phosphor samples was examined using a field emission-type scanning electron microscope (SEM) (S-4800, Hitachi, Japan).

3. RESULTS AND DISCUSSION

3.1. Crystal Structure and Microstructure Investigations. Figure 1 shows the powder XRD patterns of Ca9(Y1−xCe3+x)(PO4)7 (CYP:Ce3+y; 0 < y ≤ 1), Ca9(Y1−xGd3+y)(PO4)7 (CYP:Gd3+y; 0 < y ≤ 1), and Ca9(Y1−xPr3+z)(PO4)7 (CYP:Pr3+z; 0 < z ≤ 1); all of these patterns agree well with that of the JCPDS file no. 00-046-0402. These results indicate that the Ca9Y(PO4)7 structure is retained and has not generated any X-ray detectable impurity to appear even when Y3+ ions are substituted by Ce3+, Gd3+, or Pr3+ ions. Ca9Y(PO4)7 crystallizes as a rhombohedral unit cell with space group R3c (No. 161), lattice constants a = 10.4442 Å and c = 37.324 Å, cell volume = 3525.89 Å3, and Z = 6. The Y3+ ion is six coordinate, and the ionic radius of six-coordinated Y3+ is 0.9 Å. However, the ionic radii of six-coordinated Ce3+, Gd3+, and Pr3+ ions are 1.01, 0.938, and 0.99 Å, respectively. On account of the matching of ionic radii and isovalent substitution, the Ce3+, Gd3+, and Pr3+ ions are expected to occupy the Y3+ ions sites in the Ca9Y(PO4)7 host. Shown in Figure 2 are the distribution of particle size and SEM images of Ca9Y(PO4)7:0.2Ce3+, Ca9Y(PO4)7:0.7Gd3+, and Ca9Y(PO4)7:0.2Pr3+ phosphors. The particle size distribution of UVR phosphors was found to vary from 1 to 35 μm, and the average particle size of Ca9Y(PO4)7:0.2Ce3+, Ca9Y(PO4)7:0.7Gd3+, and Ca9Y(PO4)7:0.2Pr3+ phosphors are about 14.3, 12.9, and 13.4 μm. The inset of Figure 2 shows SEM micrographs of the UVR phosphors, which reveals that particles are aggregated and the shape is irregular.

3.2. Photoluminescence and Energy Level Diagrams. Figures 3 and 4 show the PL/PLE spectra and proposed energy level schemes of Ca9Y(PO4)7, Ca9Y(PO4)7:Ce3+, Ca9Y(PO4)7:Gd3+, and Ca9Y(PO4)7:Pr3+ phosphors. Figure 3a shows the excitation and emission spectra of the Ca9Y(PO4)7 host; as shown in this figure, broad absorption and emission bands at around 120–230 and 300–600 nm, respectively, are attributed to the self-emission of the Ca9Y(PO4)7 host. Under 225 nm excitation,
Ca$_9$Y(PO$_4$)$_7$:Ce$^{3+}$ phosphors emit UVA light in the form of one broad emission band centered at 346 nm, owing to the 5d$^1$ → 4f$^1$ transition of Ce$^{3+}$ ions. The PLE spectra contain several bands from 180 to 350 nm. These bands centered at 203, 225, 245, 260, and 288 nm are attributed to the crystal field splitting of Ce$^{3+}$ 5d states and are shown in Figure 3b. Ca$_9$Y(PO$_4$)$_7$:Gd$^{3+}$ phosphors show a strong 4f$^7$ → 4f$^7$ transition and a sharp UVB emission band at around 312 nm, which is attributed to the $^6P_{7/2}$ → $^6S_{7/2}$ transition of Gd$^{3+}$ ions. There is a single broad band and four excitation bands of Ca$_9$Y(PO$_4$)$_7$:Gd$^{3+}$ between 120 and 280 nm centered at 172, 184, 196, 247, and 273 nm. These bands can be ascribed to the transitions from the ground state to the host and the transition of $^6S_{7/2}$ → $^6P_{1/2}$, $^6P_{3/2}$, $^6P_{5/2}$, and $^6P_{7/2}$, as shown in Figure 3c. The excitation spectra of Ca$_9$Y(PO$_4$)$_7$:Pr$^{3+}$ phosphors show a broad absorption band from 120 to 240 nm, which is attributed to the dipole-allowed 4f$^2$ → 4f$^1$ 5d$^1$ transition of Pr$^{3+}$ ions. The PL spectra show two broad UVC emission bands at 230–340 nm, centered at 250 and 275 nm. These bands are attributed to the 4f$^1$ 5d$^1$ → 4f$^1$ transition of Pr$^{3+}$ ions, as shown in Figure 3d. On the other hand, in the case of Ca$_9$Y(PO$_4$)$_7$:zPr$^{3+}$ phosphors, we observed a very weak set of 4f$^1$ → 4f$^1$ and a sharp emission band at 490 nm under 172 nm excitation.

Figure 3. Excitation (black line) and emission spectra (red line) of (a) Ca$_9$Y(PO$_4$)$_7$, (b) Ca$_9$Y(PO$_4$)$_7$:Ce$^{3+}$, (c) Ca$_9$Y(PO$_4$)$_7$:Gd$^{3+}$, and (d) Ca$_9$Y(PO$_4$)$_7$:Pr$^{3+}$ phosphors.

Figure 4. Energy level scheme of Ca$_9$Y(PO$_4$)$_7$:Ce$^{3+}$, Ca$_9$Y(PO$_4$)$_7$:Gd$^{3+}$, and Ca$_9$Y(PO$_4$)$_7$:Pr$^{3+}$ phosphors.

Figure 5 shows the concentration dependence of the relative PL intensity of Ca$_9$Y(PO$_4$)$_7$:xCe$^{3+}$, Ca$_9$Y(PO$_4$)$_7$:yGd$^{3+}$, and Ca$_9$Y(PO$_4$)$_7$:zPr$^{3+}$ phosphors under 172 nm excitation. When the Ce$^{3+}$, Gd$^{3+}$, or Pr$^{3+}$ dopant concentration was higher than the optimal concentration, the PL intensity was found to decrease with an increase in the Ce$^{3+}$, Gd$^{3+}$, or Pr$^{3+}$ dopant concentration. The optimal doping concentration of UVA-emitting Ca$_9$Y(PO$_4$)$_7$:Ce$^{3+}$ phosphors was found to be x = 0.2 mol, and the corresponding optimal doping concentrations of UVB-emitting Ca$_9$Y(PO$_4$)$_7$:Gd$^{3+}$ phosphors and UVC-emitting Ca$_9$Y(PO$_4$)$_7$:Pr$^{3+}$ phosphors were y = 0.7 mol and z = 0.2 mol, respectively. According to the percolation model, the concentration quenching of the compound can occur by two mechanisms: (1) the interaction between the activator ions, which results in energy reabsorption among neighboring activator ions in the rare-earth sublattice; (2) energy transfer from a percolating cluster of activator ions to killer centers.

Figure 5. Concentration dependence of the relative PL intensity of Ca$_9$Y(PO$_4$)$_7$:xCe$^{3+}$, Ca$_9$Y(PO$_4$)$_7$:yGd$^{3+}$, and Ca$_9$Y(PO$_4$)$_7$:zPr$^{3+}$ phosphors under 172 nm excitation.

Figure 6. Comparison of the PL intensity of (a) UVA of Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$ (black solid line) and BaSi$_2$O$_5$:0.05Pb$^{2+}$ (red dashed line), (b) UVB of Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$ (black solid line) and YAl$_3$(BO$_3$)$_4$:0.25Gd$^{3+}$ (red dashed line), and (c) UVC of Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ (black solid line) and LaPO$_4$:0.1Pr$^{3+}$ (red dashed line) under 172 nm light excitation.
The PL intensity of the UVA emission of Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$ (CYP:0.2Ce$^{3+}$) and BaSi$_2$O$_5$:0.05Pb$^{2+}$ (BSO:0.05Pb$^{2+}$), that of the UVB of Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$ (CYP:0.7Gd$^{3+}$) and YAl$_3$(BO$_3$)$_4$:0.25Gd$^{3+}$, and that of the UVC of Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ (CYP:0.2Pr$^{3+}$) and LaPO$_4$:0.1Pr$^{3+}$ phosphors under 172 nm excitation are compared in Figure 6. The emission spectra and optimal doping activator concentrations of the above UVR phosphors are excited at 172 nm. The PL spectra of UVR-emitting Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$ show two broad emission bands at 230–340 nm, centered at 250 and 275 nm. The PL spectra of LaPO$_4$:0.1Pr$^{3+}$ show two broad emission bands in the range of 210–280 nm, centered at 227 and 260 nm; these bands are attributed to the transition of Pr$^{3+}$ ions from 5d to 4f levels. The luminescence intensity of the UVC-emitting Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ is 1.5 times that of the UVC emission of LaPO$_4$:0.1Pr$^{3+}$.

3.3. Thermal Luminescence Quenching Properties. Temperature dependence is an extremely important parameter for mercury-free fluorescent lamp applications. Figure 7 and Table 1 illustrate the temperature-dependent relative emission intensities of UVA-emitting Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$ and BaSi$_2$O$_5$:0.05Pb$^{2+}$ phosphors (excited at 225 nm and emission monitored at optimal wavelength), UVB-emitting Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$ and YAl$_3$(BO$_3$)$_4$:0.25Gd$^{3+}$ phosphors (excited at 274 nm and monitored at optimal wavelength), and UVC-emitting Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ and LaPO$_4$:0.1Pr$^{3+}$ phosphors (excitation at 220 nm and monitored at optimal wavelength). The relative emission intensity decreases with an increase in temperature from 25 to 250 °C. We observed only a 5.1% decay of UVA-emitting Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$ (9% decay for BaSi$_2$O$_5$:0.05Pb$^{2+}$) phosphors at 200 °C, an 8.8% decay of UVB-emitting Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$ (81.6% decay for YAl$_3$(BO$_3$)$_4$:0.25Gd$^{3+}$) phosphors, and a 7.6% decay of UVC-emitting Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ (9.9% decay for LaPO$_4$:0.1Pr$^{3+}$) phosphors, as shown in Figure 7a–c. These results show that UVR phosphors Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$, Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$, and Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ have excellent thermal quenching stability than BaSi$_2$O$_5$:0.05Pb$^{2+}$, YAl$_3$(BO$_3$)$_4$:0.25Gd$^{3+}$, and LaPO$_4$:0.1Pr$^{3+}$ phosphors. Fabrication of mercury-free xenon excimer discharge lamps using UV-emitting Ca$_9$Y(PO$_4$)$_7$:Ln$^{3+}$ (Ln = Ce, Gd, Pr) phosphors described in this research is currently in progress and will be reported later.

4. CONCLUSIONS

Three series of new UVR phosphors Ca$_9$Y(PO$_4$)$_7$:Ln$^{3+}$ (Ln = Ce, Gd, Pr) were synthesized by a high-temperature solid-state method, and their photoluminescence spectra, emission efficiency, and thermal stability were investigated. Ultraviolet-emitting Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$ (CYP:0.2Ce$^{3+}$) and LaPO$_4$:0.1Pr$^{3+}$ (CYP:0.2Pr$^{3+}$) phosphors, the PL intensity of Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ (CYP:0.2Pr$^{3+}$) and LaPO$_4$:0.1Pr$^{3+}$ phosphors have excellent emission efficiency and decent thermal stability. Under 172 nm excitation, the luminescence intensity of the UVA-emitting Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$ was found to be 0.3675 times that of the UVA emission of BaSi$_2$O$_5$:0.05Pb$^{2+}$; the luminescence intensity of the UVB-emitting Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$ is 1.7 times that of the UVB emission of YAl$_3$(BO$_3$)$_4$:0.25Gd$^{3+}$; and the luminescence intensity of the UVC-emitting Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ is 1.5 times that of the UVC emission of LaPO$_4$:0.1Pr$^{3+}$. The thermal stability of the above phosphors revealed that the extent of luminescence intensity decay was found to be only 9.2%, 18.2%, and 10.3% for Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$, Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$, and Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ at 250 °C, respectively. These results indicate that Ca$_9$Y(PO$_4$)$_7$:Ln$^{3+}$ (Ln = Ce, Gd, Pr) phosphors may serve as promising UVR phosphors for use in xenon excimer discharge lamps.

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Table 1. Thermal Luminescence Quenching Data for Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$, BaSi$_2$O$_5$:0.05Pb$^{2+}$, Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$, YAl$_3$(BO$_3$)$_4$:0.25Gd$^{3+}$, and LaPO$_4$:0.1Pr$^{3+}$ Phosphors

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Figure 7. Temperature-dependent PL intensity of (a) Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$ and BaSi$_2$O$_5$:0.05Pb$^{2+}$, (b) Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$ and YAl$_3$(BO$_3$)$_4$:0.25Gd$^{3+}$, and (c) Ca$_9$Y(PO$_4$)$_7$:0.2Pr$^{3+}$ and LaPO$_4$:0.1Pr$^{3+}$ phosphors.

Table 1. Thermal Luminescence Quenching Data for Ca$_9$Y(PO$_4$)$_7$:0.2Ce$^{3+}$, BaSi$_2$O$_5$:0.05Pb$^{2+}$, Ca$_9$Y(PO$_4$)$_7$:0.7Gd$^{3+}$, YAl$_3$(BO$_3$)$_4$:0.25Gd$^{3+}$, and LaPO$_4$:0.1Pr$^{3+}$ phosphors.
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