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Ceramics International 39 (2013) 9709–9713

CERAMICS INTERNATIONAL

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Short communication

Enhanced photoluminescence of $BaAl_2S_4:Eu^{2+}$ phosphor by Mg^{2+} ions doping

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Received 23 March 2013; received in revised form 10 April 2013; accepted 12 April 2013
Available online 20 April 2013

Abstract

A series of $Ba_{1-x}Mg_xAl_2S_4:0.10Eu^{2+}$ ($0.00 \le x \le 0.15$) phosphors was synthesized under vacuum in sealed silica tubes. The effects of Mg^{2+} doping on the structure and photoluminescence (PL) properties of $BaAl_2S_4:Eu^{2+}$ were investigated systematically. When Ba^{2+} was substituted by 6 mol% Mg^{2+}, the PL intensity of the resulting $Ba_{0.84}Mg_{0.06}Al_2S_4:0.10Eu^{2+}$ phosphor was approximately 2.5 times stronger than that of the single-doped $BaAl_2S_4:0.10Eu^{2+}$ phosphor under 395 nm excitation. The fluorescence lifetime of Eu^{2+} in $Ba_{0.84}Mg_{0.06}Al_2S_4:0.10Eu^{2+}$ was calculated as 0.133 μs. Commission Internationale de l'Eclairage chromaticity coordinates showed that the phosphor emissions are in the blue region. The synthesized phosphors show great promise for future applications in white light emitting diodes.

Keywords: Luminescence; Thioaluminate; Eu^{2+}; Phosphor

1. Introduction

Ternary compounds of $M^{II}M_2^{III}(S,Se)_4$ doped with rare-earth ions have been studied for several years. Such compounds are very attractive for use in lighting and display applications, such as field emission displays, thin-film electroluminescence (TFEL), and phosphor-converted light emitting diodes (LEDs) [1–6]. $SrGa_2S_4:Eu^{2+}$ has a wide excitation band that extends into the blue region; it is applicable as a green phosphor for white LEDs in which blue LEDs are used to excite the phosphor [7,8]. $ZnGa_2S_4:Eu^{2+}$ and $SrGa_2S_4:Sn,Re$ (Re = Ce, Gd) have also been studied as possible phosphors for white LEDs [9,10]. The structural and luminescence properties of $Ca_{1-x}Sr_x(Ga_{1-y}Al_y)_2S_4:Eu^{2+}$ phosphors were previously described by Yu et al. [11]. Changing the values of x and y in the phosphors causes nearly linear emission peak shifts, thereby allowing continuous tuning of the emission peak from 496 nm ($SrAl_2S_4:Eu^{2+}$) to 556 nm ($CaGa_2S_4:Eu^{2+}$). All of these phosphors can meet the application requirements of GaN-based LEDs.

In 1992, Le Thi et al. [12] systematically studied Eu^{2+}-doped $MS–Al_2S_3$ (M = Ca, Sr, Ba) systems and noted that the $BaAl_2S_4:Eu^{2+}$ phosphor has high efficiency for blue emission. Miura [13] demonstrated a high luminance blue TFEL device with a new blue-emitting $BaAl_2S_4:Eu^{2+}$ electroluminescent (EL) phosphor prepared by two target pulse-electron-beam evaporation; this EL device has a relatively high brightness of 65 cd/m^2. Blue-emitting $BaAl_2S_4:Eu^{2+}$ phosphors with suitable color coordinates, adequately high luminance, and emission efficiency allow the fabrication of full-color EL display devices. A successful prototype of such a device for commercial application was demonstrated by iFire, a company that developed a pilot industrial process to produce an EL full-color 34-inch flat-panel display [14]. This thick dielectric EL display technology features a color-by-blue scheme that uses a single high-luminescence blue phosphor in combination with green and red color conversion pigments, thereby allowing fabrication

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http://dx.doi.org/10.1016/j.ceramint.2013.04.041

of a high-definition TV display with a brightness approaching 1000 cd/m^2.

A number of studies have reported that addition of cations, such as Mg^{2+}, can help enhance the luminescence efficiency of some phosphors and that its radius matches the size limit of solid solutions [15–20]. Daisuke Fukuda et al. [15] prepared Mg-doped $BaTiO_3$:Pr^{3+} phosphors by a solid-phase reaction. The optimal Mg-doping concentration in this phosphor was 2.0 mol%. Ryu et al. [16] illustrated that the emission spectrum of a $Mg_xCa_{1-x}Al_2O_4$:Eu^{2+},Nd^{3+} phosphor with a Mg content of $x=0.25$ shows maximum intensity. Yajun Lu [19] reported that Mg^{2+} doping remarkably enhances the emission and absorption intensity of $CaSi_2O_2N_2$:Eu^{2+} phosphor. Yong-Kwang Jeong [20] also reported that partial replacement of Sr^{2+} with Mg^{2+} in $(Zn_{0.6}Sr_{0.4})_2Ga_2S_5$:0.20$Eu^{2+}$ significantly improves the luminescence intensity of the phosphor by about 1.6 times than its original intensity.

In this study, we thus attempt to introduce Mg^{2+} ions in $BaAl_2S_4$:Eu^{2+} phosphors and expect to improve the emission intensity of phosphors. We prepared a series of Mg-substituted $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} ($0.00\leq x\leq 0.15$) phosphors by sealed vacuum quartz-tube synthesis. Characterization techniques, including powder X-ray diffraction (XRD), photoluminescence (PL) spectra, PL decay curves, and color coordinates (x,y), were undertaken to investigate the optical properties of the resulting $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} phosphors.

2. Experimental

The starting sulfide materials, BaS and EuS, were pre-prepared by a solid-state reaction method in horizontal tube furnaces at high temperature. BaS was prepared from $BaCO_3$ [analytical reagent (A.R.) grade] under flowing H_2S gas at 1000 °C for 2 h. EuS was prepared from Eu_2O_3 (99.99%) in a CS_2 reducing atmosphere at 1200 °C for 3 h. Stoichiometric amounts of BaS, Mg (A.R.), Al (A.R.), EuS, and 25 mass% excess S (A.R.) were thoroughly mixed, placed in quartz ampoules (150 mm length, 10 mm inner diameter), sealed, evacuated to 1×10^{-6} Torr, and then fired at 980 °C for 5 h.

The structure of the final products was examined by XRD using a Bruker D8 ADVANCE X-ray diffractometer with Cu Kα radiation at 40 kV and 40 mA. XRD patterns were collected at a scan rate of 10°/min in the range of $10°\leq 2\theta\leq 70°$. The PL, PL excitation spectra and decay curve of the $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} phosphors were recorded using an FLS920 combined fluorescence lifetime and steady state spectrometer (Edinburgh Instruments) equipped with a 450 W Xe lamp and a 150 W nF900 nanosecond flash lamp with a pulse width of 1 ns and a pulse repetition rate of 40–100 kHz.

3. Results and discussion

Structurally, $BaAl_2S_4$ crystallizes in a cubic structure, space group $\bar{3}Pa$(T6h), with a lattice parameter $a=12.65$ Å [12]. Fig. 1 presents the XRD patterns of the $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} (where $x=0.00$, 0.03, 0.06, 0.08, 0.10, and 0.15) phosphors. No impurity phases, such as BaS, MgS, Al_2S_3, or Al_2O_3, were detected, which are in good agreement with JCPDS Card no. 76-1054. The lattice constants of the phosphors were calculated as $a=12.60$ Å and $V=2003.38$ Å^3 for $BaAl_2S_4$:0.10Eu^{2+}, consistent with the unit-cell parameters reported in the literature [12]. The ionic radii (r) of Ba^{2+}(1) (CN=6), Ba^{2+}(2) (CN=12), and Al^{3+} (CN=4) are 135 pm, 161 pm, and 39 pm, respectively. The radii of ions of the dopant elements, Mg^{2+} (CN=6, $r=72$ pm) and Eu^{2+} (CN=6, $r=117$ pm) are such that these dopant ions were expected to occupy the Ba^{2+} sites in the $BaAl_2S_4$ host due to the close radii and identical valence of the ions.

Fig. 2 presents the excitation and emission spectra of the $BaAl_2S_4$:0.10 Eu^{2+} sample. The excitation spectrum (curve a) extends over a broad range of wavelengths. $BaAl_2S_4$ has a band gap of 4.1 eV [21]. The high-energy band (250–320 nm) is assigned to host absorption from the valence band to the conduction band and the low-energy bands (320–450 nm) are

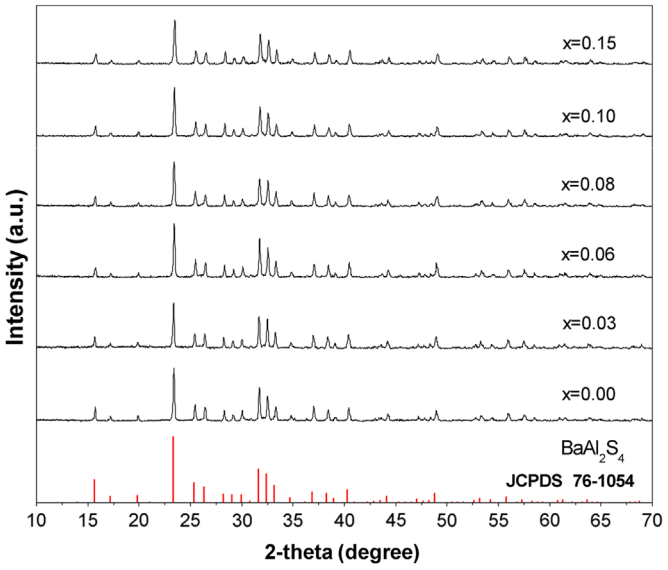

Fig. 1. XRD patterns of $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} with varying Mg^{2+} concentrations ($x=0.00$, 0.03, 0.06, 0.08, 0.10, and 0.15).

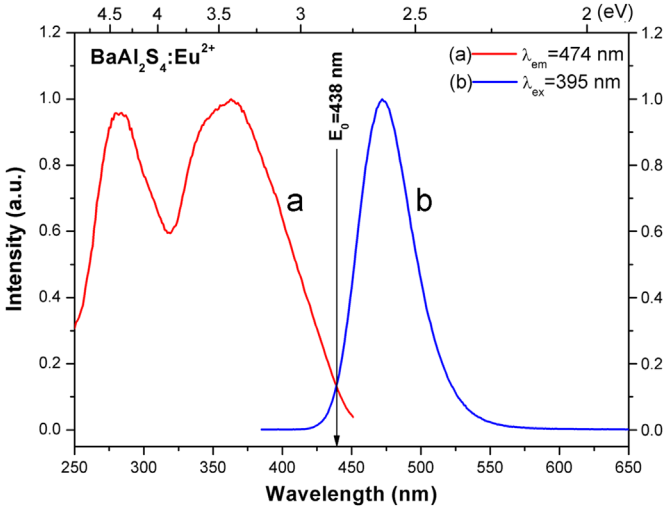

Fig. 2. (a) Excitation ($\lambda_{em}=474$ nm) and (b) emission ($\lambda_{ex}=395$ nm) spectra of $BaAl_2S_4$:0.10Eu^{2+}.

due to $4f^7(^8S_{7/2}) \rightarrow 4f^6(^7F)5d^1$ transitions of the Eu^{2+} ion. The $BaAl_2S_4$:0.10Eu^{2+} phosphor shows a blue emission band peaking at 474 nm under 395 nm excitation (curve *b*). The full width at half maximum (FWHM) of the emission spectrum of the phosphor is 44 nm. Compared with the common value of FWHM of Eu^{2+} ions in most phosphors, which is about 50–100 nm, the values in our case are smaller, which indicates weak interactions between Eu^{2+} ions and the host material. In the lighting field, decreases in the FWHM of the emission band are helpful in increasing the luminous output of the phosphor [22]. No emission was observed from BaS:Eu^{2+} at 570 nm, which may be expected if unreacted BaS:Eu^{2+} compound is present in the system [23]. No other special line peaks of Eu^{3+} were observed in the emission spectrum (curve *b*), which proves that Eu^{3+} ions in the matrix crystals are completely reduced to Eu^{2+}. The $BaAl_2S_4$:Eu^{2+} emission is ascribed to the dipole-allowed transition from the lower $4f^6(^7F)5d^1$state to the $4f^7(^8S_{7/2})$ ground state of Eu^{2+} ions. In $BaAl_2S_4$, and Ba^{2+} ions reside in sites of two different symmetries. Eight Ba^{2+} ions are in six-fold coordination with point symmetry $S_3(3)$, while four Ba^{2+} ions, in positions *a* and *b*, are in 12-fold coordination with point symmetry $S_6(3)$ [24]. Eu^{2+} ions are capable of substituting both types of Ba^{2+} ions in $BaAl_2S_4$. Although different Ba sites are present in the phosphor, the $BaAl_2S_4$:Eu^{2+} powder shows only one emission band. This behavior has previously been observed in homologous thioaluminates [12,25] and thiogallate phosphors [4].

The energies of f–d absorption and d–f emission can be written according to the formalism of Dorenbos [26] as

$$E_{abs} = E_{free} - D \text{ and } E_{emi} = E_{free} - D - \Delta s \quad (1)$$

where E_{free} is the energy difference between the lowest $4f^7$ level and the $4f^6(^7F_0)5d$ level for free or gaseous ions, *D* is the energy lowering, also called the red shift, and Δs is the Stokes shift. Given that the excitation spectrum is not well resolved, the position of the lowest 5d excited level of Eu^{2+} (E_{abs}) is generally estimated by using the mirror-image relationship between the emission and excitation spectra [21,27]. The Stokes shift (Δs) can be estimated as twice the energy difference between the zero-phonon line and the energy of the emission maximum. The position of the zero-phonon line is considered the intersection point of the excitation and emission spectra. In the present case, E_{abs} is about 3.08 eV (402 nm) and the Δs was calculated as 0.23 eV (3778 cm^{-1}). This value is comparable with the Δs reported for $BaGa_2S_4$:Eu^{2+} (4000 cm^{-1}) [28]. Knowledge of the value of E_{abs} allows us to determine the *D* of the f–d transition with respect to the free ion (E_{free}=4.19 eV for Eu^{2+}) [29]. We found $D = E_{free} - E_{abs} = 4.19 - 3.08 = 1.11$ eV (8940 cm^{-1}), which is weaker than that 1.3 eV in $BaGa_2S_4$:Eu^{2+} compound [27]. The difference of bond covalence is the main reason for this behavior. Due to the smaller size of the Al cation, Al–S bonds are shorter and more covalent than Ga–S ones. The replacement of Ga by Al leads to a decrease of the covalence of the Ba–S bond and consequently to the decrease of the nephelauxetic effect. The weaker red shift explains the higher energy of the f→d and d→f transitions for $BaAl_2S_4$:Eu^{2+} compared to $BaGa_2S_4$:Eu^{2+} compound.

Fig. 3 shows the emission spectra of $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} at various Mg^{2+} contents (x=0.00, 0.03, 0.06, 0.08, 0.10, and 0.15) obtained under 395 nm excitation; the dependence of PL intensity on Mg^{2+} concentration is also illustrated. The emission peak of the Mg^{2+} ions slightly shifted toward a longer wavelength in $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} as the Mg^{2+} concentration increased. The emission peak position shifted from 474 nm to 476 nm as the Mg^{2+} content increased from 0.00 mol% to 6 mol% respectively. At Mg^{2+} concentration of 15 mol%, the emission peak position shifted to 477 nm. The red shift observed can be attributed to enhancements in the crystal field strength surrounding the Eu^{2+} ions. The PL intensity increases with increasing Mg^{2+} content until it reaches a maximum at x=0.06, after which it falls steadily with further increases in Mg^{2+} content. When the doping concentration of Mg^{2+} is 6 mol%, the obtained optimal PL intensity of $Ba_{0.84}Mg_{0.06}Al_2S_4$:0.10$Eu^{2+}$ is approximately 2.5 times stronger than that of the single-doped $BaAl_2S_4$:0.10Eu^{2+} phosphor under 395 nm excitation. The results indicate that doping a certain amount of Mg^{2+} ions into $BaAl_2S_4$:Eu^{2+} phosphor can efficiently enhance the luminescent intensity of this phosphor. It can be explained that the incorporation of Mg^{2+} ions cause some change in the field strength surrounding the Eu^{2+} ions. Further increasing Mg^{2+} contents over 6 mol% cannot effectively enhance the luminescence intensity of the phosphors. Excessive doping may bring about several defects that can affect the efficiency of energy transfer from the host matrix to the activated Eu^{2+} ions [30].

The fluorescence decay time of Eu^{2+} in the $Ba_{0.84}Mg_{0.06}Al_2S_4$:0.10$Eu^{2+}$ phosphor with optimal composition was measured at room temperature. Fig. 4 shows that the emission decay curves of the Eu^{2+} ions are fitted well by a second-order exponential equation:

$$I(t) = A_1\exp(-t/\tau_1) + A_2\exp(-t/\tau_2) \quad (2)$$

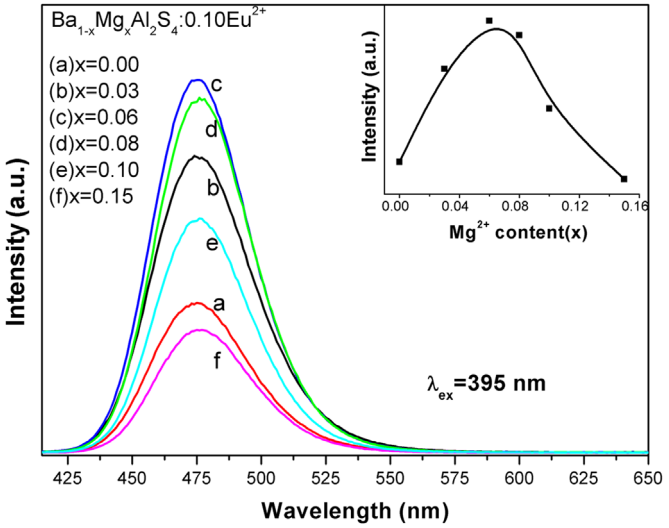

Fig. 3. Emission spectra of $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} with varying Mg^{2+} concentrations: (a) x=0.00, (b) x=0.03, (c) x=0.06, (d) x=0.08, (e) x=0.10, and (f) x=0.15. Inset shows the influence of Mg^{2+} content on the emission intensity of the $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} phosphors (λ_{ex}=395 nm).

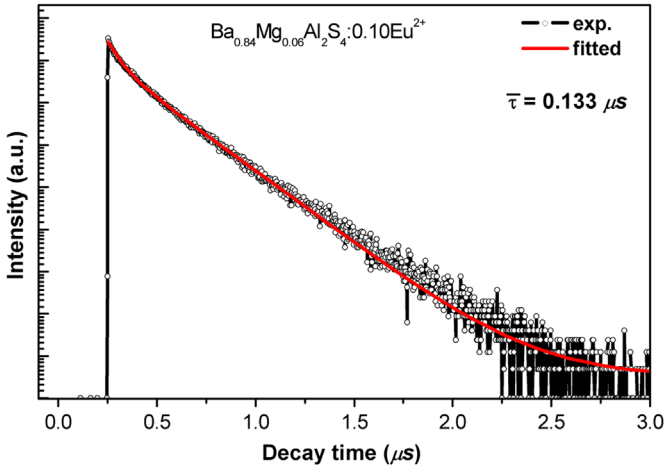

Fig. 4. Decay curve of $Ba_{0.84}Mg_{0.06}Al_2S_4$:0.10$Eu^{2+}$ at room temperature (λ_{em}=476 nm, λ_{ex}=395 nm).

where I is the luminescence intensity, t is the time, τ_1 and τ_2 are the slow and fast components of the decay lifetimes, respectively, and A_1 and A_2 are their corresponding fitting parameters. On the basis of Eq. (2), the lifetimes are determined to be 0.290, and 0.067 μs, which are reasonable and there are two different alkali earth cation sites in $BaAl_2S_4$ lattice. The average lifetime of Eu^{2+},$\overline{\tau}$, could be calculated from Eq. (3) [31]:

$$\overline{\tau} = \frac{A_1\tau_1^2 + A_2\tau_2^2}{A_1\tau_1 + A_2\tau_2} \quad (3)$$

The value of τ was calculated as 0.133 μs. This result is reasonable for the allowed 5d–4f transition of Eu^{2+} ions, which is known occur in less than a microsecond [32]. The result also shows that the lifetime is short enough for potential applications in displays and lights.

Fig. 5 shows the Commission Internationale de l'Eclairage (CIE) 1931 chromaticity coordinates of $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} (x=0.00, 0.03, 0.06, 0.08, 0.10, and 0.15) calculated from the emission spectra in Fig. 3. The colors of all the $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} phosphors are located in the blue region. As the concentration of Mg^{2+} in $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} is increased, the CIE coordinates change from the value of (0.119,0.155) for x=0.00 to the value of (0.117,0.165) for x=0.15. According to the slight red shift of the emission peak center of the PL spectra in Fig. 3, the chromaticity of Mg^{2+} codoped $BaAl_2S_4$:0.10Eu^{2+} phosphors changes slightly as a result of their increased Mg^{2+} concentrations. Magnified views of the phosphors are shown in the inset of Fig. 5. The color coordinates of $BaMgAl_{10}O_{17}$:Eu^{2+} (BAM) are better than those of $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} upon color saturation. The small variation in color coordinates observed shows that the as-synthesized phosphors have good color stability.

4. Conclusions

A series of $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} (x=0.00, 0.03, 0.06, 0.08, 0.10, and 0.15) phosphors is synthesized under vacuum in sealed silica tubes. XRD studies confirm a cubic structure with

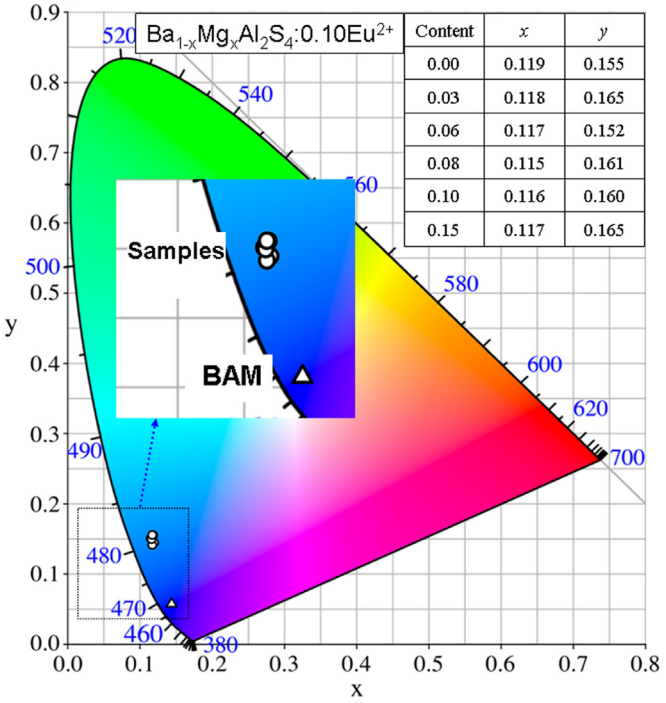

Fig. 5. Color coordinates of BAM and $Ba_{1-x}Mg_xAl_2S_4$:0.10Eu^{2+} (x=0.00, 0.03, 0.06, 0.08, 0.10, and 0.15)

space group $\overline{3}Pa$(T6h). The excitation and emission spectra of the phosphors are broadband due to the $4f^7$–$4f^65d^1$ transitions of Eu^{2+}. Doping of the $BaAl_2S_4$:Eu^{2+} phosphor with appropriate amounts of Mg^{2+} ions can enhance its luminescence intensity. When Ba^{2+} is substituted by 6 mol% Mg^{2+}, the $Ba_{0.84}Mg_{0.06}Al_2S_4$:0.10$Eu^{2+}$ phosphor exhibits about 2.5 times stronger PL intensity than the single-doped $Ba_{0.84}Mg_{0.06}Al_2S_4$:0.10$Eu^{2+}$ phosphor under 395 nm excitation. The fluorescence lifetime of Eu^{2+} is calculated as 0.133 μs for $Ba_{0.84}Mg_{0.06}Al_2S_4$:0.10$Eu^{2+}$. CIE chromaticity coordinates show that the phosphor emissions are in the blue region. Since the phosphor excitation band lies in the near-UV excitable region, which gives blue emission, it can be used for applications in near-UV phosphor-converted white LED lighting and display devices.

Acknowledgments

This research was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (Nos. 2012055455 and 012R1A1A2003979). A $BaAl_2S_4$:Eu^{2+} phosphor, supplied by the Display and Lighting Phosphor Bank at Pukyong National University. This work was also supported by the National Natural Science Foundations of China (Grant no. 21201141), and the Chinese Universities Scientific Fund (Grant no. QN2011119).

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