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ARTICLE TYPE

Effect of Gd/La substitution on the phase structures and luminescence properties of (La,Gd)Sr₂AlO₅:Ce³⁺ solid solution phosphors

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A series of yellow-emitting $La_{0.975-x}Gd_xSr_2AlO_5:0.025Ce^{3+}$ phosphors have been synthesized by a solid-state reaction. All the samples retain the same tetragonal crystal structure and the phosphors emit the yellow emission with a peak maxima shifting

- ¹⁰ from 540 nm to 556 nm depending on different Gd/La ratio when excited by 440 nm blue light, and their temperature dependence photoluminescence, fluorescence decay curves, and CIE values were also discussed. With increasing Gd/La ratio, the red-shift behaviors can be ascribed to the enhancing covalence, and the ¹⁵ emission intensities firstly increased, then decreased, and
- maximized at x = 0.6. A white light-emitting diode (w-LED) lamp was fabricated based on the selected yellow-emitting La_{0.375}Gd_{0.6}Sr₂AlO₅:0.025Ce³⁺ phosphors and combining a 460 nm blue-emitting InGaN chip. The packaged w-LED lamp gave
- ²⁰ CIE chromaticity coordinates of (0.3562, 0.3660) with a warm color temperature of 4664 K, a color rendering index of 83.

1 Introduction

Among the phosphors developed in the past decades, silicates and aluminates based phosphors have drew much attentions ²⁵ owing to their excellent luminescence properties, superior chemical stability, cheap resources and easy preparation, which could be adopted to different applications including the long lasting phosphorescence phosphors (SrAl₂O₄:Eu²⁺,Dy³⁺) white light emitting diodes (w-LEDs) phosphors (Y₃Al₅O₁₂:Ce³⁺), and ³⁰ so on.^{1,2} Except for the above mentioned merits, silicates/aluminates based phosphors also possess more chances

- for the tunable luminescence properties since the chemical compositions, crystal structures and activators in the host can be relatively easy to be modified via the controlled experiments.³⁻⁴
- ³⁵ For example, the fabrication of the iso-structural solid solution phosphors open a new avenue for the discovery of the color-tunable phosphors, and some interesting silicates/aluminates phosphors have been also explored in our group, such as, M_xMg₂Al_{4+x}Si_{5-x}O₁₈:Eu^{2+,5} La₅(Si_{2+x}B_{1-x})(O_{13-x}N_x):Ce^{3+,6} Di (Ca, Al) O (Ca^{3+,7} and ca an

40 $Bi_2(Ga,Al)_4O_9:Cr^{3+},^7$ and so on.

Herein, we will discuss a kind of aluminates solid solution phosphors $(La,Gd)Sr_2AlO_5:Ce^{3+}$. As we know, the yellowemitting phosphor LaSr_2AlO_5:Ce^{3+} (LSA:Ce) was firstly reported by Im et al.⁸ They discovered that LSA:Ce with proper excitation

⁴⁵ at 450 nm and emission at 556 nm was potential for w-LEDs package. Also, they also developed a series of isostructural

LaSr₂AlO₅-based solid solution phosphors, such as Sr₃AlO₄F:Ce³⁺, (Sr₃AlO₄F-Sr₃SiO₅):Ce³⁺, (Sr₃SiO₅-La₂SrAlO₅):Ce³⁺, and so on.⁸⁻¹¹ From a general view, the 50 structural relationship of this kind of silicates/aluminates based phosphors and effect on the luminescence properties have been investigated, however, the optimization of the chemical composition of the LaSr₂AlO₅:Ce³⁺ was not reported till now. Therefore, we focused on the structural evolution of 55 (La,Gd)Sr₂AlO₅ solid solutions. We investigated the Gd/La ratio dependent (La,Gd)Sr₂AlO₅ phase structures, PL properties and thermal stability of Ce3+-doped phosphors, and their related luminescence mechanisms. As for La0.975-xGdxSr2AlO5:0.025Ce3+ (x = 0.0.975) solid solutions phosphors, there is a red-shift 60 tendency for the photoluminescence after Gd³⁺ replacing La³⁺ under the excitation of 440 nm. A w-LEDs lamp was fabricated based on the selected yellow-emitting La0.375Gd0.6Sr2AlO5:0.025Ce3+ phosphors and combining a 460 nm blue-emitting InGaN chip, and the luminescence properties 65 was also evaluated in this paper.

2 Experimental section

2.1 Materials and synthesis

La0.975-*x*Gd_xSr₂AlO₅:0.025Ce³⁺ (x = 0, 0.2, 0.4, 0.6, 0.8 and 0.975) samples were synthesized by a high temperature solid-⁷⁰ state reaction. The starting materials of La₂O₃ (99.9%), Gd₂O₃ (99.9%), SrCO₃ (99.9%), Al₂O₃ (99.9%) and SrF₂ (99.9%) were all purchased from the Beijing Chemical Company, China, and CeO₂ (99.995%) were supplied by China Minls (Beijing) Research Institute, Beijing, China. The raw materials were ⁷⁵ weighed and mixed in the given stoichiometric proportion, and then ground by an agate mortar. Subsequently, the mixtures were placed into an alumina crucible, and then fired in a reducing atmosphere of H₂/N₂ (5%/95%) for 4 h between 1350 °C and 1400 °C depending on different La/Gd ratios. For the samples ⁸⁰ with x = 0 and 0.2, the sintering temperature is fixed at 1350 °C, and 1400 °C is need for the pure phase formation when the Gd contents *x* are 0.4, 0.6, 0.8 and 0.975, respectively.

2.2 Characterization

X-ray diffraction (XRD) data were obtained on an X-ray spowder diffraction (Philips X'PertPW-3040) with Cu Ka radiation ($\lambda = 0.15406$ nm) at 40 kV and 35 mA over the angular range of 10°≤20≤70°. Room-temperature excitation and emission

spectra were measured on a fluorescence spectrophotometer (F-4600, HITACHI, Japan) with a photomultiplier tube operating at 400 V, and a 150 W Xe lamp used as the excitation light source. The temperature-variable luminescence properties were measured

- 5 on the same device, which was coupled with a self-made heating attachment and a computer-controlled electric furnace. The decay curves were obtained from an Edinburgh FLSP920 Fluorescence Spectrophotometer spectrophotometer. Prototype white LED devices were fabricated by applying a homogeneous mixture of
- 10 50%/50% by weight of the vellow La0.375Gd0.6Sr2AlO5:0.025Ce3+ phosphors and transparent silicone resins on blue InGaN LEDs $(\lambda_{max} = 460 \text{ nm})$. The electroluminescent (EL) spectrum, color temperature, color rendering index (CRI), and CIE value of the LEDs were measured and evaluated by an Everfine PMS-80 Plus 15 UV-Vis-near IR Spectro-photocolorimeter system.

Results and discussion 3

3.1 Phase formation and crystal structure

Fig. 1(a) displays the results of XRD patterns of a series of asprepared La0.975-xGdxSr2AlO5:0.025Ce3+ phosphors. The crystal 20 structure of LaSr2AlO5 is isostructural with the standard file of EuSr₂AlO₅ (JCPDS no.70-2197, tetragonal, space group I4/mcm).¹² It can be clearly seen that the prepared La0.975xGdxSr2AlO5:0.025Ce3+ samples were well-matched with the diffraction peaks of EuSr₂AlO₅, indicating that the phase 25 structures of these compounds keep unchanged upon the doping of Ce³⁺ or La³⁺/Gd³⁺ substitution. This result indicates that continuous solid solution phase in the whole range can be obtained by the solid-state reaction method, suggesting the good structural compatibility.



Fig. 1. (a) XRD patterns of as-prepared $La_{0.975-x}Gd_xSr_2AlO_5:0.025Ce^{3+}$ (x = 0,0.2,0.4,0.6,0.8,0.975) samples. (b) Amplifying XRD patterns between 30° and 32° , and the standard data for EuSr₂AlO₅ (JCPDS card 70-2197) 35 are shown as the reference.

Fig. 1(b) shows the enlarged XRD patterns between 30° and 32° for this series of samples, which obviously displays the peak positions shift to large diffraction angle with the increasing amounts of Gd³⁺ ions content. According to Bragg equation: ⁴⁰ $2dsin\theta = n\lambda$, where d represents interplanar distance, θ represents the angle between the incident rays and the corresponding crystallographic plane, n is diffraction series, λ represents wavelength. When more La3+ ions (0.116 nm) replacing with Gd3+ (0.105 nm), d value gradually becomes small, therefore θ value 45 subsequently becomes large, in accordance with the shift to large

diffraction angle. In order to clearly display the quantitative

variation of these phosphors, the Fig. 2 shows the lattice constants of La_{0.975-x}Gd_xSr₂AlO₅:0.025Ce³⁺ phosphors, which are calculated with the XRD data. An obvious decrease in the lattice 50 constant 'a', 'c' and 'V' for the as-prepared La0.975-_xGd_xSr₂AlO₅:0.025Ce³⁺ samples was observed with the increase of the Gd³⁺ ions content, also indicating that the solid solution phosphors La_{0.975-x}Gd_xSr₂AlO₅: $0.025Ce^{3+}$ exist in the all x range of 0-0.975, while the same crystal structure is retained.¹³



Fig. 2. Variation of lattice parameters and cell volume of La_{0.975}. $_x$ Gd_xSr₂AlO₅:0.025Ce³⁺ as a function of x, which is obtained from the corresponding XRD data: (a) a, (b) c, and (c) cell volume V.

In order to make certain the structure of them, Fig. 3 displays 60 the schematic unit cell diagram and the evolution of this series of $La_{0.975\text{-}x}Gd_xSr_2AlO_5{:}0.025Ce^{3+}.$ As reported previously, 8 Sr has two sites named Sr1 and Sr2. The 8h site is occupied by Sr (Sr1), which could be also occupied by La or Gd . The 4a site is occupied by Sr (Sr2). The 4b site is only occupied by Al. The 4c 65 site and 16l site are occupied by O, labeled as O1 and O2, respectively, and Fig. 3 clearly demonstrated such a structural occupation. Moreover, the structure can be regarded as layers of (La/Gd/Sr1) and O1 alternating with layers of and Sr2 and $[Al(O2)_4]$ tetrahedra. La/Gd and Sr1 has 8(2 + 6) coordination 70 with O1 and O2. Sr2 has 10(2 + 8) coordination with respect to O1 and O2.14 The polyhedral geometry of these two sites are depicted alongside the crystal structural description. Therefore, we can find the excellent structural compatibility in such a stable phase, and the Gd³⁺/La³⁺ substitution will form the continuous 75 solid solution phases.



Fig. 3. The schematic structure diagram of (La,Gd)Sr₂AlO₅:0.025Ce³⁺ suggesting the isostructural evolution depending on the La3+/Gd3+ substitution.

3.2 Luminescent Properties of Ce^{3+} doped (La,Gd)Sr₂AlO₅ phosphors

The room temperature excitation and emission spectra of $La_{0.975-x}Gd_xSr_2AlO_5:0.025Ce^{3+}$ (x = 0-0.975) phosphors are ⁵ displayed in Fig. 4(a). All PLE spectra consist of two distinct bands, which is resulting from the f-d transition of Ce³⁺. Besides, all PL spectra consist of a broad yellow band from 455 to 700 nm with a maximum wavelength at about 540-550 nm, which is typically ascribed to the lowest excited state 5d¹ to the ground

- ¹⁰ states ${}^{2}F_{5/2}$ and ${}^{2}F_{7/2}$ transition of the Ce³⁺ ion.^{15,16} At the same time, the emission bands of all the samples have a gradual redshift trend from 540 nm to 556 nm when the Gd³⁺ substitution increases from x = 0 to x = 0.975, which can be obviously shown in the normalized spectra in Fig. 4(b). The red-shift behavior in
- $_{15}$ La_{0.975-x}Gd_xSr_2AlO_5:0.025Ce^{3+} system could attribute to the variation of the crystal-field strength. Generally, the crystal field splitting (Dq) is written as follows: 17

$$Dq = \frac{Ze^2r^4}{R^5} \tag{1}$$

where Z is the valence or charge of the anion, e is the charge of ²⁰ an electron, r is the radius of the d wave function and R is the distance between the central ion and its ligands. When La³⁺ ion is substituted by the smaller Gd³⁺ ion, leading to shorter interatomic distance, in other words, the crystal field splitting (Dq) is larger. Consequently the emission wavelength of these phosphors move

- ²⁵ to lower energy region up to 556 nm at x = 0.975. Such a behavior has been observed in many other system, such as the emission spectrum of Gd₃Al₅O₁₂:Ce³⁺ compared with that of Y₃Al₅O₁₂:Ce³⁺. In addition, It can be also obviously illustrated that the highest emission intensity was obtained at x = 0.6, which
- ³⁰ was about 30% improved in comparison with the sample x = 0. When the Gd content (*x*) was larger than 0.8, the emission intensity was rapidly reduced. This result can be explained by the different crystalline behaviours. As given in the experimental details, the higher sintering temperature (1400 °C) is needed in ³⁵ order to obtain the pure phases of La_{0.975-x}Gd_xSr₂AlO₅:0.025Ce³⁺
- phosphors (x = 0.4, 0.6, 0.8 and 0.975) owing to the complex chemical compositions. It is accepted that the increased temperature will help to increase the crystalline of the powder particles, so that the emission intensities can be improved with
- ⁴⁰ enhanced crystalline behaviours and big powder particles, and such a result has been also found in this similar system by other group.¹³ However, the emission intensities can't be further improved for the increased Gd content since the temperature keeps invariable for these samples, so that the composition at x =
- ⁴⁵ 0.6 may possess the most appropriate size for the luminous.



Fig. 4. (a) The excitation and emission spectra of $La_{0.975}$. _xGd_xSr₂AlO₅:0.025Ce³⁺ (x = 0, 0.2, 0.4, 0.6, 0.8 and 0.975). (b) The ⁵⁰ normalized emission spectra,.

Fig. 5 presented decay curves of the Ce³⁺ emission in the La_{0.975-x}Gd_xSr₂AlO₅:0.025Ce³⁺ phosphors under excitation at 440 nm, monitored at corresponding emission peaks in different compositions. All luminescent decay curves could be fitted well ⁵⁵ with double-exponential function by the following equation:^{18,19}

$$I = A_{1} \exp(-t/\tau_{1}) + A_{2} \exp(-t/\tau_{2})$$
(2)

where *I* is the luminescent intensity and A_1 and A_2 are constants; *t* represents time, and τ_1 and τ_2 are the decay times for the exponential components. Using these parameters, the average ⁶⁰ decay time τ can be calculated by the following formula:

$$\tau = (A_1 \tau_1^2 + A_2 \tau_2^2) / (A_1 \tau_1 + A_2 \tau_2)$$
(3)

Therefore, the values of average decay times of the Ce³⁺ emission of La_{0.975-x}Gd_xSr₂AlO₅:0.025 Ce³⁺ phosphors (x = 0, 0.2, 0.6, 0.8 and 0.975) are determined to be 32.99, 38.68, 36.23, 38.27 and

⁶⁵ 37.02 ns, respectively. It can be seen that decay time become longer when replacing La³⁺ ion with Gd³⁺ ion. The influence of the compressed Ce³⁺ polyhedral is ascribed to interaction of the neighboring La³⁺/Gd³⁺ ions, the increasing concentration of Gd³⁺ will in turn increase the distance of the activators, then the energy ⁷⁰ transfer possibility among the activators will decrease, so that we can find the increasing lifetimes values in this series of La_{0.975-x}Gd_xSr₂AlO₅:0.025 Ce³⁺ phosphors with increasing Gd content and invariable Ce³⁺ concentrations.

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Fig. 5. Decay curves of the Ce^{3+} emission in the $La_{0.975-x}Gd_xSr_2AlO_5:0.025$ Ce^{3+} phosphors under excitation at 440 nm, monitored at corresponding emission peaks wavelength.

5 3.3 Thermal stability CIE chromaticity coordinates, and the properties of the fabricated white LEDs lamp

In order to discuss the possible application of the studied phosphors, the thermal quenching behaviors of the selected phosphor have been evaluated. As we know, thermal stability for ¹⁰ phosphors used in w-LEDs is important because it has a considerable impact on the light output and CRI. Fig. 6(a), (c)

- and (e) show the temperature dependent emission spectra of typical samples in the temperature range from 25 to 200 °C. As can be seen from figures, all the emission intensities decrease ¹⁵ with increasing temperature. At the same time, all the emission peaks for the selected La_{0.975-x}Gd_xSr₂AlO₅:0.025Ce³⁺ phosphors shows a slight blue shift behavior with raising temperature, which
- can be ascribed to the thermally active phonon assisted excitation from the excited states of the lower-energy emission band to 20 higher-energy emission band in the excited states of the
- activators.²⁰ Furthermore, with the introduction of the Gd into La sites, the thermal quenching become well compared to the original one (from 22% to 40 %). The reason is that the intermediate compositions can balance the overall bond valance
- ²⁵ at the random La/Gd sites, as discovered in the Sr_{2-x}Ba_xSiO₄:Eu²⁺ system.²¹ Moreover, we have also compared the thermal quenching behaviours of the commercial YAG:Ce phosphor and the composition optimized La_{0.375}Gd_{0.6}Sr₂AlO₅:0.025Ce³⁺ phosphor in Fig. 6(g). The poor thermal stability for our present
- ³⁰ samples should be ascribed to the compositional disordering of La³⁺/Sr²⁺/Gd³⁺, and it might provide the increasing alternate quenching paths in the host lattice with the substitution of activators $Ce^{3+,22}$ To better learn more about the difference and origin of temperature-dependent luminescence behaviors, the
- ³⁵ activation energy (ΔE) was calculated using the Arrhenius equation:²³

$$I_T = \frac{I_0}{1 + c \exp(-\frac{\Delta E}{kT})} \tag{4}$$

where I_0 is the initial emission intensity of the phosphor at room temperature, I_T is the emission intensity at different temperatures, $_{40} \Delta E$ is the activation energy for thermal quenching, c is a constant, and k is Boltzmann constant (1.380650524 × 10⁻²³ J/K). We can see that emission intensity The activation energy ΔE can be calculated by plotting $\ln[(I_0/I_T) -1]$ against 1/kT, which the absolute value of the slope is the activation energy ΔE . Fig. 6 (b), (d) and (f) display the fitting lines according to Eq. (4) and the calculated activation energy ΔE values of 0.3889 eV, 0.2551 eV and 0.1903 eV respectively. The decreased values in thermal quenching property after the replacement of Gd³⁺ ions can be explained by configurational coordinate model of this system. As so shown in Fig.6(h), firstly, we mark the intersection between the ground state and excited state as L1 and L2 respectively. As we know, the Stokes shift (ΔR) becomes larger with the substitutions of Gd. As a consequence, the energy between the exited state and intersection, which is activation energy for thermal quenching, so becomes smaller. It means that electrons need less energy (lower temperature) to reach the intersection to achieve quenching. Therefore, the thermal stability becomes worse with the



Fig. 6. (a)The variable temperature spectrum chart of $La_{0.975-x}Gd_xSr_2AlO_5:0.025Ce^{3+}$ (x = 0, 0.6 and 0.975). (b)Calculations of activation energy of corresponding $La_{0.975-x}Gd_xSr_2AlO_5:0.025Ce^{3+}$ samples, according to the variable temperature data. (c) Configurational coordinate ⁶⁵ model of $La_{0.975-x}Gd_xSr_2AlO_5:0.025Ce^{3+}$

As demonstrated in the normalized emission spectra of (La,Gd)Sr₂AlO₅:0.025Ce³⁺ phosphor, red shift behaviors can be found with increasing Gd/La ratio. Therefore, the CIE chromaticity coordinates for the selected compositions of ⁷⁰ La_{0.975}Sr₂AlO₅:0.025Ce³⁺ and La_{0.375}Gd_{0.6}Sr₂AlO₅:0.025Ce³⁺

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phosphors were measured and presented in Fig. 7(a), and it is clear to find that the color tone can be tuned from greenish yellow (0.355, 0.558) to orange yellow (0.408, 0.546). As also given in the digital photographs for the phosphors irradiated by the 365

- ⁵ nm UV lamp, the as-observed emitting colour is obvious to the naked eyes. Furthermore, in order to further demonstrate the potential application of yellow-emitting (La,Gd)Sr₂AlO₅:0.025Ce³⁺ phosphor, we combined one composition-optimized yellow phosphor
- ¹⁰ (La_{0.375}Gd_{0.6}Sr₂AlO₅:0.025 Ce³⁺) with commercial blue LED chip to realize the white light. Fig. 7(b) displays the electroluminescent (EL) spectrum of a packaged w-LEDs lamp under a 25 mA forward bias current, and the photos of the asfabricated w-LEDs lamp and its actual emission under the same
- $_{15}$ forward bias are shown in the insert. The EL spectrum clearly displays a blue chip emission peak at around 460 nm and a yellow emission band corresponding to the emission peak of La_{0.375}Gd_{0.6}Sr_2AIO_5:0.025Ce^{3+} at around 550 nm. The measurement result reveals that CIE color coordinates are
- ²⁰ (0.3562, 0.3660) at a warm white light correlated color temperature (CCT) of 4664 K and a suitable CRI values (Ra) is 83. In contrast with the blue InGaN chip packaged with YAG:Ce³⁺ phosphor (Ra = 75, CCT = 7756 K), the w-LEDs in this study show lower CCT values corresponding to the warm
- $_{25}$ white light and higher Ra values.²⁴ As discussed, La_{0.975-x}Gd_xSr₂AlO₅:0.025Ce³⁺ could be a promising candidate for applications of w-LEDs.



30 Fig. 7. (a) CIE chromaticity diagram for the selected phosphors excited at 365 nm. The inset shows the digital photographs of the two representative phosphors. (b) The electroluminescence spectrum of the packed white LEDs lamp under the forward bias current of 25 mA. The insert image gives the encapsulation of the LED lamp and its illumination image.

35 4 Conclusion

In summary, we have successfully synthesized a series of La_{0.975-x}Gd_xSr₂AlO₅:0.025 Ce³⁺ phosphors by the solid state reaction, and the phase structure and luminescence properties were investigated in detail. Isostructural solid solutions of 40 (La,Gd)Sr₂AlO₅ phase can be formed in the whole range between the two end members of LaSr2AlO5 and GdSr2AlO5. Red shift behaviors can be found with increasing Gd/La ratio, which is ascribed to the strong crystal field splitting for Gd-enriched compositions. The thermal stable luminescence and decay curves 45 of this series of samples have been also studied and the composition optimized La0.375Gd0.6Sr2AlO5:0.025Ce3+ phosphor has been determined. Based on the evaluation of the as-fabricated white LEDs lamp via the selected phosphor and the commercial blue LED chip, the lower CCT values and higher Ra values show 50 that this series of phosphors would have potential use in white light-emitting diodes.

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60 Notes and references

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Effect of Gd/La substitution on the phase structures and luminescence properties of (La,Gd)Sr₂AlO₅:Ce³⁺ solid solution phosphors

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Structure, optimum composition and luminescence properties of yellow-emitting $La_{0.975-x}Gd_xSr_2AlO_5:0.025Ce^{3+}$ phosphors for white LEDs have been studied.

