

Luminescence of Sr₃Al₂O₆:Tb³⁺, Eu³⁺ Phosphor: A Strong Candidate as White Light Emitting Lamp Phosphor

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Abstract

We report here a single-phased $Sr_3Al_2O_6:Tb^{3+}$, Eu^{3+} as a candidate for white light emitting phosphor for the application in fluorescent lamps. The $Sr_3Al_2O_6:Tb^{3+}$, Eu^{3+} phosphor exhibits emission over blue, green and red region of the visible spectrum. The phosphor when excited by 254 nm shows sharp lines peaked in 400-550nm due to ${}^5D_3 {}^{-7}F_J$ transitions and ${}^5D_4 {}^{-7}F_J$ transitions of Tb^{3+} ions and sharp peaks in the orange–red region (580–700 nm) originating from ${}^5D_0 {}^{-7}F_J$ transitions of Eu^{3+} ions. The energy transfer phenomenon in $Sr_3Al_2O_6:Tb^{3+}$, Eu^{3+} system were observed as a result of increasing concentration of europium in $Sr_3Al_2O_6:Tb^{3+}$ system. Varying concentration of Tb^{3+} and Eu^{3+} are studied in detail for attaining the white light emission from this host lattice. The thermoluminescence study was performed on the β - irradiated $Sr_3Al_2O_6:Tb^{3+}$, Eu^{3+} phosphor to study its change in trap depths due to addition of europium as co-activator. From the results obtained it is suggested that $Sr_3Al_2O_6:Tb^{3+}, Eu^{3+}(0.025\%)$ can be serve as white light emitting fluorescent lamp phosphor.

Keywords: ceramics; chemical synthesis; X-ray diffraction; photoluminescence; thermoluminescence; white light emitting phosphor.

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1. Introduction

Thrust of white light emission from single host lattice in the field of lamp phosphors is attracting today's scientist [1]. The emission of white light in the case of glass using Tb^{3+} and Eu^{3+} is also reported [2]. Although the commercial FLs contains a mixture of triphosphors-the blue-emitting BaMgAl₁₀O₁₇: Eu^{2+} [4], the redemitting Y₂O₃: Eu^{3+} [5], and the green-emitting LaPO₄: Ce³⁺, Tb³⁺ [6] under UV light at 254 nm were wellestablished, the development of new phosphors continues because of the importance of phosphor efficiency required for different applications [7,8]. As we known, trivalent Tb and Eu ions, as the promising species that provide optical emission in green and red regions, have been investigated by many groups [9,10].

The $Sr_3Al_2O_6:Eu^{2+}$, Dy^{3+} has a useful application as a mechanoluminscent phosphor [11]. Strontium aluminate, due to its long light persistence property is one such material that has been studied extensively for more than a decade now [12, 13]. In our earlier work, we have reported the photoluminescence and thermoluminescence properties of rare earth doped $Sr_3Al_2O_6$ phosphor [14, 15, 16]. In this paper we study the effect of addition of Eu^{3+} in the $Sr_3Al_2O_6:Tb^{3+}$ phosphor on its optical and thermoluminescent properties. The white light emission from this system was obtained for the $Sr_3Al_2O_6:Tb^{3+}(0.025\%)$, $Eu^{3+}(0.025\%)$ phosphor. The energy transfer phenomenon was also studied by keeping the concentration of Tb^{3+} constant and varying the concentration of Eu^{3+} in the system. The effect of incorporation of co-activator in the $Sr_3Al_2O_6:Tb^{3+}$ system was also reported in the case of thermoluminescence study.

2. Experimental

2.1. Sample preparation

The synthesis of the $Sr_3Al_2O_6$ doped with rare earths has been elaborated in our previous papers [14,15]. The phosphor samples were prepared by the sol-gel reflux technique. The starting materials taken were strontium nitrate, aluminum nitrate and the rare earths in the nitrate form. All of them were acquired from S.D. Fine Chemicals with rare earths of purity 99.9%. The first set of the samples was prepared by doping the Terbium nitrate (1%) and Europium nitrate (x%) in the host matrix. The powders were weighed according to the nominal composition of $Sr(NO_3)_2$ +Al(NO₃)_3.9H₂O+ Tb.(NO₃)_3 + x Eu. (NO₃)_3(x = 0.025\%, 0.1\%, 0.5\%). Second set of samples were prepared with varying concentration of both terbium nitrate and europium nitrate.

The starting materials strontium and aluminum nitrates were taken in 1:2 proportions and dissolved in the appropriate amount of distilled water and kept for stirring. Thereafter the rare earths compounds with different concentrations were mixed to the above solution. Citric acid and ethylene glycol were added to the solution after one hour of constant stirring. The resulting gel was set for refluxing at 100°C for 3 hours. The gel thus obtained, was kept for drying in an oven maintained at 100°C for 10 hours. The yellowish gel then transformed into a fluffy material. It was then kept for firing at a temperature maintained at 900°C in a furnace for 16 hours in air. The white powder obtained on firing, was ground using an agate mortar and pestle and was then subjected to various characterizations.

2.2. X-ray diffraction, Photoluminescence and Thermoluminescence Characterization

Phase identification of the powders was carried out by the X-ray powder diffraction using RIGAKU D'MAX III Diffractometer having Cu K α radiation. The photoluminescence (Emission and Excitation spectra) were recorded at room temperature using spectrofluorophotometer RF-5301 of SHIMADZU make with a xenon source. The slit width of both the excitation and the emission monochromator was kept at 1.5nm and the sensitivity was kept high for all the samples. The thermoluminescence glow curve of all the samples were recorded using Nucleonix-1009 make thermoluminescence reader at a constant heating rate of 2.0°C/s. The amount of sample used for studying TL was 5mg and was irradiated with β rays from a Sr-90 source of 50 mCi strength.

3. Result and Discussion

3.1. X-ray diffraction studies

In order to determine the crystal structure and phase purity of the phosphors, X-ray diffraction (XRD) analysis was carried out. Figure – 1 shows X-ray diffraction (XRD) pattern of the $Sr_3Al_2O_6$:Tb³⁺(1%), Eu³⁺ (0.1%)phosphor. From the XRD pattern analysis it was found that the prominent phase formed was $Sr_3Al_2O_6$. The $Sr_3Al_2O_6$ phase belongs to the space group pa3 having a cubic crystalline structure [17]. The traces of other phases of SrO-Al₂O₃ system were very weak. As the XRD patterns are similar for all the terbium and europium concentrations, only one with 1% Tb³⁺ and 0.1% Eu³⁺ has been shown and it is matches very well with that reported by Kennedy et al. [18].



Figure 1: XRD pattern of $Sr_3Al_2O_6$: Tb³⁺ (1%), Eu³⁺ (0.1%)

3.2 Photoluminescence of $Sr_3Al_2O_6$: Tb^{3+} , Eu^{3+} Phosphor

Figure 2 shows the excitation and emission spectra of the $Sr_3Al_2O_6$:Tb³⁺, Eu³⁺ system when excited with the 254nm wavelength. The excitation spectra were measured for 545 nm of Tb³⁺(1%) emission. All the samples

show excitation nearly same except increase in the intensity of the 390nm peak of Eu³⁺ transition on increasing the Eu³⁺ concentration. Hence only two excitation spectra are represented in the figure. The curve A shows the emission pattern of Tb³⁺ (1%) having narrow band emission at 489, 545, 585 and 625 nm for the characteristic multiplet transition of ${}^{5}D_{4} \rightarrow {}^{7}F_{J=6,5,4,3}$ respectively [19]. In particular, the highest sharp line peaked at 542nm is characteristic of ${}^{5}D_{4} \rightarrow {}^{7}F_{J=6,5,4,3}$ respectively [19]. In particular, the highest sharp line peaked at 542nm is characteristic of ${}^{5}D_{4} \rightarrow {}^{7}F_{J=6,5,4,3}$ respectively [19]. In particular, the highest sharp line peaked at 542nm is characteristic of ${}^{5}D_{4} \rightarrow {}^{7}F_{5}$ of Tb³⁺ 4f-4f transitions. Curve B shows the emission spectra of Sr₃Al₂O₆:Tb(1%),Eu(0.025%). The intensity of the terbium emission band decreases slightly and the characteristic emission of trivalent Eu is observed at 580 nm, 592 nm, 595 nm, 613 nm, 617 nm ascribed to ${}^{5}D_{0} \rightarrow {}^{7}F_{J}$ (J = 0, 1, 2) multiplet transitions [20]. Hence this indicates the possibility of energy transfer occurs between Tb³⁺ and Eu³⁺ ions in the host lattice. To observe further the energy transfer phenomenon, we slowly increased the concentration of the Eu³⁺ in the system by keeping the concentration of terbium constant i.e. 1%.



Figure 2: Excitation and Emission characteristics of Sr₃Al₂O₆:Tb³⁺ (1%), Eu³⁺(x%).

For emission the excitation wavelength = 254 nm Curve A = $Sr_3Al_2O_6$: Tb(1%); B = $Sr_3Al_2O_6$:Tb(1%),Eu(0.025%); C = $Sr_3Al_2O_6$:Tb(1%),Eu(0.1%); D = $Sr_3Al_2O_6$:Tb(1%),Eu(0.5%); E = $Sr_3Al_2O_6$:Tb(1%),Eu(1%); F = $Sr_3Al_2O_6$:Eu(1%).

The concentration of europium was increased from 0.025% to 1%. The emission spectra of this double doped system signifies that the emission intensity of terbium decreases nonlinearly and Eu³⁺ increases linearly with increase in europium concentration. The emission spectra of $Sr_3Al_2O_6$:Tb³⁺ (1%), Eu³⁺ (1%) i.e., curve E shows a beautiful yellowish white emission can be obtained from this phosphor when excited with the ultraviolet wavelength (254 nm). The corresponding energy levels scheme of Tb³⁺ and Eu³⁺ and the possible optical transition involved in the energy transfer processes are schematically depicted in Fig. 3. Out of all these emissions we observed only emissions shown in Figure 2. The ⁵D₃ transition of Tb³⁺ is already absent due to the cross relaxation at higher concentration and the Eu³⁺ shows only the transition ⁵D₀ \rightarrow ⁷F_J (J = 0, 1, 2). When Tb³⁺ ions absorb UV light, the excitation energy could be released not only by emitting green light but also by

transferring some of its emission energy to Eu^{3+} ions, which finally exhibits a yellowish white emission of Tb^{3+} and Eu^{3+} ions.



Figure 3: Expected PL spectra and Schematic representation of the energy transfer in Sr₃Al₂O₆: Tb³⁺,Eu³⁺.

3.3 Photoluminescence excitation spectra of $Sr_3Al_2O_6$: Tb^{3+} , Eu^{3+} phosphor at varying concentration of Terbium and Europium

Thus from the above PL emission spectra of $Sr_3Al_2O_6:Tb^{3+},Eu^{3+}$, the probabilities of obtaining white light emission from the single host lattice increased. Hence in order to attain this prospect the concentrations of Tb and Eu were varied randomly so as to get the uniform emission in all over visible range of spectra.

Figure 4 shows the excitation spectra of the $Sr_3Al_2O_6$:Tb³⁺, Eu³⁺ phosphor measured at different emission wavelengths for the respective concentration of Tb³⁺ and Eu³⁺.

For curve E the excitation spectra was measured for 594 nm. The excitation band show peak around 254 nm which shows the emission is due to the transition of charge transfer band for the system.

Curve D shows the sharp excitation at 254 nm which show a small split at 253 nm when measured for the emission wavelength 545 nm.

The excitation peak of Curve A, B and C shows the band around 254 nm. One can observe that except for the $Sr_3Al_2O_6$:Tb³⁺(0.025%), Eu³⁺(0.025%) system, all the systems shows a weak excitation peak at around 390nm, which is the characteristic f–f transition line of Eu³⁺ which is attributed to the transitions from ⁷F₀ ground state to ⁵L₆ level.

All the emission spectra were measured for the 254 nm excitation wavelength. The PL spectrum yields multiemission for $Sr_3Al_2O_6:Tb^{3+}$ (0.025%), Eu^{3+} (0.025%) i.e., Curve E shows peaks at 414, 430, 436, 468, 487, 496, 509, 545, 580, 592, 595,613 and 617 nm wavelength corresponding to the ⁵D₃ and ⁵D₄ transitions of Tb³⁺ as well as ${}^{5}D_{0}$ to ${}^{7}F_{1,2}$ transitions of Eu³⁺. Only this system shows all the possible transitions of Tb³⁺ and Eu³⁺ in this system. The white light emission is also possible in this system when doped with appropriate amount of activator and co-activator.



Figure 4: Excitation and emission spectra of Sr₃Al₂O₆:Tb³⁺,Eu³⁺.

$$\begin{split} \text{Curve } A &= Sr_3Al_2O_6\text{:}Tb(0.1\%)\text{,}Eu(1\%)\text{;}\\ B &= Sr_3Al_2O_6\text{:}Tb(0.025\%)\text{,}Eu(1\%)\text{;}\\ C &= Sr_3Al_2O_6\text{:}Tb(0.5\%)\text{,}Eu(0.025\%)\text{;}\\ D &= Sr_3Al_2O_6\text{:}Tb(0.5\%)\text{,}Eu(0.1\%)\text{;}\\ E &= Sr_3Al_2O_6\text{:}Tb(0.025\%)\text{,}Eu(0.025\%)\text{,} \end{split}$$

The emission spectra for curve D shows the peaks at 468, 482, 544, 552, 592, 612 and 616 nm when excited with the 254 nm wavelength. The transition from the ${}^{5}D_{3}$ level of Tb³⁺ is not clear but the main emission peak of Tb³⁺ (545 nm) becomes prominent in this phosphor. Also the emission of Eu³⁺ in the region 592, 612 and 616 has also increased. The reason behind the distortion of ${}^{5}D_{3}$ emission in this phosphor is may be due to the increase in the concentration of Tb³⁺ which results in cross-relaxation of the luminescence [21]. It is well-known that the ${}^{5}D_{0} \rightarrow {}^{7}F_{1}$ transition of Eu³⁺ belongs to the magnetic-dipole transition which scarcely changes the crystal field strength around the Eu³⁺ ions and this transition is independent of the symmetry and the site occupied by Eu³⁺ ions in the host. While the transition of ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$ belongs to a forced electric dipole transition and its intensity is very sensitive to the site symmetry of the Eu³⁺ ions [22,23]. As shown in Fig. 4, the ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$ electric-dipole transition dominates the emission spectrum, which suggests that the Eu³⁺ is located in an asymmetric cation environment.

The emission spectra of curve C also show all the possible transition of Tb^{3+} whereas as the Eu^{3+} concentration is low the intensity of its emission is also feeble. From this phosphor it is observed that the low concentration of Eu^{3+} improves the ${}^{5}D_{3}$ emission of Tb^{3+} . The Sr₃Al₂O₆:Tb³⁺(0.1%), Eu³⁺ (1%) shows emission at 545, 551, 593, 611 and 617 nm. The high concentration of Eu³⁺ dominates the emission over Tb³⁺ and hence for emission in all over visible region one has to control the Eu³⁺ concentration in this system.

It is observed from the above study that when the co-activator concentration i.e. Eu^{3+} concentration is as low as, 0.025%, the ${}^{5}D_{3}$ transition of Tb^{3+} becomes possible and the emission in the blue region is observed. The addition of small amount of Eu^{3+} in the $Sr_{3}Al_{2}O_{6}$: Tb^{3+} can forbid the cross-relaxation process of the Tb^{3+} .

The above experimental results exhibit that the emission of the Tb^{3+} and Eu^{3+} doped $Sr_3Al_2O_6$ phosphor is located at around blue, green and red area, respectively, suggesting that tricolor (blue, green and red) emitting bands may be obtained in a single host lattice.

3.4 Thermoluminescence (TL) Characteristics



Figure 5: Thermoluminescence glow curve of Sr₃Al₂O₆:Tb³⁺ (1%),Eu³⁺ (x%) phosphor

Curve A =
$$Sr_3Al_2O_6$$
:Eu(1%);
B = $Sr_3Al_2O_6$:Tb(1%);
C = $Sr_3Al_2O_6$:Tb(1%),Eu(0.025%);
D = $Sr_3Al_2O_6$:Tb(1%),Eu(0.1%);
E = $Sr_3Al_2O_6$:Tb(1%),Eu(0.5%).

The energy transfer phenomenon in the $Sr_3Al_2O_6:Tb^{3+},Eu^{3+}$ phosphor can also be observed using the thermoluminescence characteristics. The phosphors were subjected to the thermally stimulated luminescence (TL) studies to ascertain the defects present in the synthesized compounds. Figure 5 shows the thermoluminescence glow curve of the $Sr_3Al_2O_6:Tb^{3+},Eu^{3+}$ phosphor, where the concentration of Eu^{3+} gradually increases whereas the concentration of Tb^{3+} kept constant at 1%. The test dose of 100 Gy from a β source (Sr-90) of 50 mCi strength was given to all samples. Earlier we reported the dosimetric study of the $Sr_3Al_2O_6:Tb^{3+}$ phosphor [15]. For comparison the glow curve of $Sr_3Al_2O_6:Eu^{3+}$ (1%) is also shown in the figure. The curve A

shows the single low intensity peak at around 215 °C, whereas the curve B shows peaks around 124, 164 and 340 °C. The addition of Eu³⁺ in Sr₃Al₂O₆:Tb³⁺ (1%) changes the glow curve pattern completely. The thermoluminescence glow curve C shows the peaks around 124, 164 and 262 °C. The intensity of the 124 and 164 °C peaks also decreases by small amount. A new peak at 262 °C is observed on addition of Eu³⁺. The well defined and prominent peak of 340 °C changes into the shoulder of the 262 °C peak. The 262 °C peak shows the formation of new traps due to incorporation of Eu³⁺ in Sr₃Al₂O₆:Tb³⁺ phosphor. The glow curve D shows that the peak of 124 and 164 °C has been reduced remarkably and the TL intensity of the 262 °C peak increases with small intensity. Further increase in the Eu³⁺ concentration to 0.5% doesn't affect much to the intensity of 124 and 164 °C peaks as it has already low intensity, but it helps to improve the intensity of the 262 °C peak. This suggests that the after addition of Eu³⁺ in the Sr₃Al₂O₆:Tb³⁺ phosphor the tendency of the charges getting trapped at 124 and 164 °C peaks reduces and they managed to get trapped and new trapping level which is further deep at 262 °C. The 262 °C is also not stable and reduces drastically within 48 hours. Also this peak consists of very complicated structure having more than 3-4 small glow curves which was tough to isolate for calculation of TL parameters of this system.

3.5 Thermoluminescence glow curve of $Sr_3Al_2O_6$: Tb^{3+} , Eu^{3+} phosphor with varying concentration of Tb^{3+} and Eu^{3+}



Figure 6: Thermoluminescence glow curve of Sr₃Al₂O₆:Tb,Eu phosphor

Where
$$T = Tb^{3+}$$
 and $E = Eu^{3+}$

The TL response of different concentration of Tb^{3+} and Eu^{3+} on the TL glow curve of $Sr_3Al_2O_6$: Tb^{3+} , Eu^{3+} has been studied above room temperature. Untill now the TL parameters of SRA-Al₂O₃ system were calculated to know the trap depth responsible for phosphorescence. But in our earlier work, we have reported the new aspect

of this system as a thermoluminescent dosimeter [15]. The TL measurement of five sets of varying concentration of Tb^{3+} and Eu^{3+} were performed as shown in the figure 6. The test dose of 100 Gy from a β -source (Sr-90) of 50 mCi strength was given to all samples. The TL glow curve of Sr₃Al₂O₆:Tb³⁺ (0.025%), Eu³⁺ (0.025%) shows peak around 124, 164 and 340 °C with very high intensity. Also the TL dosimetric peak 164 °C of this phosphor shows all the characteristics of the dosimetric peak and shows less fading when stored in dark for around three months. The TL peaks of above phosphors were isolated and the TL parameters were calculated as in the case of earlier work [15]. The analysis of this glow curve was done and the order of kinetics was calculated by measuring the geometrical factor:

$$\mu_{g} = \delta/\omega \tag{1}$$

The values of the δ, ω and τ were calculated as follows:-

 $\delta = T_2 - T_m$, The high-temperature half width of the glow curve.

 $\omega = T_2 - T_1$, The full width of the glow peak at its half height.

 $\tau = T_m - T_1$, The low- temperature half width of the glow curve.

From the values of the geometrical factor it is clear that all the three peaks obey General Order Kinetics. The trap depth or the activation energy was calculated using the Chen's equation [24]

$$E\alpha = C\alpha(E_{\alpha} = c_{\alpha}(kT_{m}^{2}/\nu) - b_{\alpha}(2kT_{m})$$
⁽²⁾

Where k = Boltzmann constant, $T_m = Peak$ Temperature

The value of the constants, $c_{\alpha} \& b_{\alpha}$ was calculated by the Chen's Equation for the General Order Kinetics. Where α was replaced by δ , ω or τ as the case may be.

Table 1: The list of calculated values by the Chen's equation.

Peak	Geometrical	Eτ	E_{ω}	E _δ	Mean E
Temperature	Factor (μ_g)				
		(ev)	(ev)	(ev)	(ev)
$T_m(^{\circ}C)$					
124	0.55	0.75	0.75	0.746	0.748
163	0.5	0.77	0.79	0.81	0.79
350	0.475	0.65	0.71	0.77	0.71

The $Sr_3Al_2O_6:Tb^{3+}$ produces deep traps at temperature around 120 to 350 °C which could be useful for dosimetric purpose due to their thermal stability and low fading [15]. Whereas in the case for

 $Sr_3Al_2O_6:Tb^{3+}(0.025\%)$, $Eu^{3+}(0.025\%)$ increases the TL intensity of the dosimetric peak which points to role of the co-dopant in the host matrix is to increase the overall oxygen vacancies and in consequence number of trapped charge carriers that in turn induce an increment of the TL signal intensity. The increase in the intensity of glow peaks of the phosphor indicates that the presence of the europium ions increases the TL efficiency by increasing the number of trapping charge carriers and the radiative recombination efficiency of those freed electrons or holes by thermally stimulating them. Evidently, the co-dopant ion increases the defect formation that promotes the trapping and recombination processes [25,26].

The PL spectra of this phosphor also show emission in all ranges of the visible region. Hence this phosphor can be considered as a good candidate for white light emitting lamp phosphor as well as dosimetric purpose.

It has been observed from the TL glow curve of rest of the $Sr_3Al_2O_6$:Tb(x%), Eu(y%) phosphors that increase in the Eu³⁺concentration is responsible for decrease in the intensity of 124, 164 and 340 °C peak. The glow curves of $Sr_3Al_2O_6$:Tb(0.025%), Eu(1%) and $Sr_3Al_2O_6$:Tb(0.1%), Eu(1%) shows the effect of increase in the Tb³⁺ concentration on the glow curve. As per the figure the increase in the Tb³⁺ concentration reduces the intensity of the 262 °C glow peak.

4. Conclusion

From all the above results and discussion it can be conclude that the $Sr_3Al_2O_6$:Tb³⁺ (0.025%), Eu³⁺ (0.025%) shows emission in the blue, green and red region of the visible spectra which project it as a strong candidate for white light emitting lamp phosphor. The TL characteristics of this phosphor also show its application in the field of dosimetry.

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