Research Article

Effect of Eu^{3+} Concentration on Luminescence Studies of $\text{Y}_4\text{Al}_2\text{O}_9$ Phosphor

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The present paper reports the effect of europium concentration on photoluminescence (PL) and thermoluminescence (TL) studies of Eu^{3+} doped $\text{Y}_4\text{Al}_2\text{O}_9$ phosphor using inorganic materials like yttrium oxide (Y_2O_3), aluminium oxide (Al_2O_3), boric acid (H_3BO_3) as a flux, and europium oxide (Eu_2O_3). The sample was prepared by the modified solid state reaction method, which is the most suitable for large-scale production. The prepared phosphor sample was characterized using X-ray diffraction (XRD), field emission gun scanning electron microscopy (FEGSEM), Fourier transform infrared spectroscopy (FTIR), photoluminescence (PL), thermoluminescence (TL), and CIE techniques. The PL emission was observed in the range of 467, 535, 591, 611, 625, and 629 nm for the $\text{Y}_4\text{Al}_2\text{O}_9$ phosphor doped with Eu^{3+} (0.1 mol% to 2.5 mol%). Excitation spectrum was found at 237 and 268 nm. Sharp peaks were found around 591, 611, and 625 nm with high intensity. From the XRD data, using Scherer's formula, the calculated average crystallite size of Eu^{3+} doped $\text{Y}_4\text{Al}_2\text{O}_9$ the phosphor is around 55 nm. Thermoluminescence study was carried out for the phosphor with UV irradiation. The present phosphor can act as single host for red light emission in display devices.

1. Introduction

During the past decades, nanostructured materials have attracted considerable attention for their novel and enhanced properties; for example, the Mn doped ZnS phosphor can yield both high luminescent efficiencies and short lifetime [1, 2]. Nanostructured materials may be developed to form a novel type of luminescent materials for display applications.

The Y_2O_3 - Al_2O_3 system is a promising material for refractory coatings and for ceramic and semiconductor processing technology [3, 4]. Doped yttrium aluminium garnet (YAG) is widely used as a laser host material [5, 6], and yttrium aluminum perovskite (YAP) is used as scintillation host material [7]. In addition, rare earth doped YAG is also employed as a phosphor [8, 9]. However, there are few reports on rare earth doped $\text{Y}_4\text{Al}_2\text{O}_9$ (YAM). It has been reported that the space group for the crystal structure of $\text{Y}_4\text{Al}_2\text{O}_9$ is P21/c of monoclinic system [10, 11]. The Al atoms are coordinated to four oxygen atoms, the Y atoms

are coordinated to either six or seven oxygen atoms [11], and its site symmetry is C_1 [12]. There are four formula units in the unit cell of the room temperature phase of $\text{Y}_4\text{Al}_2\text{O}_9$ and four different rare earth sites in the asymmetric unit. Xia et al. [13] have synthesized the $\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}^{3+}$ phosphor through a sol-gel combustion method, where the $\text{Y}_4\text{Al}_2\text{O}_9$ phase can form through sintering at 800°C. However, higher doping concentration could be realized in $\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}^{3+}$ nanocrystal host lattice. Liu and Su have also prepared the $\text{Y}_4\text{Al}_2\text{O}_9:(\text{Tb}^{3+}, \text{Eu}^{3+})$ by sol-gel process [14]. D.-Y. Wang and Y.-H. Wang [15] have studied the VUV excitation and photoluminescence characteristics of this $\text{Y}_{3.8}\text{Al}_2\text{O}_9:\text{Re}_{0.2}$ ($\text{Re} = \text{Tb}^{3+}, \text{Eu}^{3+}$) phosphor synthesized via a citric-gel method. This phosphor shows strong absorption in VUV region. The results indicate that this phosphor could be one of the potential candidates for PDPs applications.

In this paper, a new modified solid state reaction method was used to synthesize $\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}^{3+}$ phosphor. This process

perfectly combines the merits of solid state reaction processing and a high-temperature combustion process. This synthesis has the advantages of inexpensive precursors, convenient process control, and large mass production. The $Y_4Al_2O_9:Eu^{3+}$ phosphor was synthesized at a high temperature of $1300^\circ C$. The structure, morphology, photoluminescence, and thermoluminescence study of $Y_4Al_2O_9:Eu^{3+}$ phosphor is investigated in detail.

2. Synthesis and Characterization

To prepare $Y_4Al_2O_9$ with various concentrations of europium (0.1 mol% to 2.5 mol%), consisting, heating in stoichiometric amounts of reactant mixture are taken in alumina crucible and fired in air at $1000^\circ C$ for 1 hour in a muffle furnace. Every heating is followed by intermediate grinding using agate mortar and pestle. The Eu^{3+} activated $Y_4Al_2O_9$ phosphor was prepared via high temperature modified solid state diffusion. The starting materials were as follows: Y_2O_3 , Al_2O_3 , Eu_2O_3 , and H_3BO_3 (as a flux) in molar ratio (0.1% to 2.5% of Eu) were used to prepare the phosphor. The mixture of reagents was ground together to obtain a homogeneous powder. After being ground thoroughly in stoichiometric ratios by using an agate mortar by dry grinding for nearly 45 minutes, to ensure the best homogeneity and reactivity, powder was transferred to alumina crucible and then heated in a muffle furnace at $1300^\circ C$ for 4 hours [16]. The phosphor materials were cooled to room temperature naturally.

The sample was characterized using XRD, FTIR, EDX (energy dispersive X-ray analysis) FEGSEM, and HRTEM. The XRD measurements were carried out using Bruker D8 Advance X-ray diffractometer. The X-rays were produced using a sealed tube and the wavelength of X-ray was 0.154 nm (Cu K-alpha). The X-rays were detected using a fast counting detector based on Silicon strip technology (Bruker LynxEye detector). Observation of particle morphology was investigated by FEGSEM (field emission gun scanning electron microscope) (JEOL JSM-6360). The photoluminescence (PL) emission and excitation spectra were recorded at room temperature by use of a Shimadzu RF-5301 PC spectrofluorophotometer. The excitation source was a xenon lamp. Thermally stimulated luminescence glow curves were recorded at room temperature by using TLD reader I1009 supplied by Nucleonix Sys. Pvt. Ltd. Hyderabad [17–19]. The obtained phosphor under the TL examination is given UV radiation using 254 nm UV source. Heating rate used for TL measurement is $6.7^\circ C s^{-1}$.

3. Results and Discussion

The XRD pattern of the sample is shown in Figure 1. The width of the peak increases as the size of the particle decreases. The size of the particle has been computed from the full width half maximum (FWHM) of the intense peak using Debye Scherer formula. Particle size of sample in the range 55 nm is found. Formula used for calculation is

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

FIGURE 1: PXRD pattern of Eu^{3+} doped $Y_4Al_2O_9$ phosphor (1.5% Eu).

Here D is particle size, β is FWHM (full width half maximum), λ is the wavelength of X ray source, θ is angle of diffraction, and $D = 0.9 * 1.54 / 0.252 * \cos(29.33) = 55$ nm.

For XRD pattern corresponding miller indices values are calculated which match with JCPDS card no. as shown in Figure 1. Sample show orthorhombic structure. The crystallite size calculated using Debye Scherer formula is 55 nm.

3.1. FEGSEM Micrographs. Figures 2(a)–2(e) show FEGSEM micrographs recorded for different resolutions of prepared sample, which indicates clearly that the particles are of uniform size having good connectivity with grain. The average particles have shown nanoimages with the particle size 25–55 nanometre.

3.2. HRTEM Image. Figures 3(a)–3(c) show the HRTEM images of prepared phosphor. Here, HRTEM results show the formation of nanoparticles of prepared sample and satisfactory results with PXRD and FEGSEM results.

3.3. Energy Dispersive X-Ray Spectroscopy. It is an elemental analysis of prepared phosphor. Figure 4 shows the element Y, Al, O, and Eu present in the sample. It is supporting data for formation of $Y_4Al_2O_9$ phosphor.

3.4. FTIR Analysis. FTIR spectrum of $Y_4Al_2O_9:Eu$ powder is shown in the Figure 5. This spectrum expresses a strong peak at 457 cm^{-1} and 723 cm^{-1} which are the characteristics of Y–O vibrations [20]. This spectrum shows a broad band at 3440 cm^{-1} which is due to the OH^- stretching vibrations of free and hydrogen-bonded hydroxyl groups [21]. The peak found at 766 cm^{-1} is probably because of Al–O vibrations [22]. Presence of B–O gives rise to an IR peak within a range of $1326\text{--}1415\text{ cm}^{-1}$; we also have a peak at 1464 cm^{-1} , so we predict this peak as a result of B–O vibration because we have used H_3BO_3 as flux [22, 23]. All these discussed peaks found together confirm the formation of $Y_4Al_2O_9$ phosphor.

FIGURE 2: ((a)–(e)) FEGSEM image of prepared phosphor with different resolutions (1.5% Eu).

FIGURE 3: ((a)–(c)) HRTEM image of prepared phosphor with different resolutions (1.5% Eu).

FIGURE 4: EDX of Eu^{3+} doped $\text{Y}_4\text{Al}_2\text{O}_9$ phosphor (1.5% Eu).

FIGURE 5: FTIR spectra of $Y_4Al_2O_9:Eu$ doped phosphor.

FIGURE 6: PL excitation spectra of $Y_4Al_2O_9:Eu$ (0.1%) doped phosphor excited with 612 nm.

3.5. Photoluminescence Study. Figures 6 and 8 shows the excitation spectra of $Y_4Al_2O_9:Eu^{3+}$ phosphor. The excitation spectra of $Y_4Al_2O_9:Eu^{3+}$ phosphor mainly consist of the charge transfer and (CTB) of Eu^{3+} located in 220–400 nm. The CTB of crystalline phosphor shows blue shift. The energy position of Eu^{3+} CTB is closely related to the covalency of $Eu-O$ bond and the coordination number of Eu^{3+} . The covalency of $Eu^{3+}-O^{2-}$ bond is strongly influenced by the next nearest cation M^{3+} ($M = Y^{3+}, Al^{3+}$). Stronger chemical bond between $Eu-O$ bond and M^{3+} ions forms for the crystalline phosphor, compared with amorphous one. Therefore, the CTB of crystalline $Y_4Al_2O_9:Eu^{3+}$ is located at shorter wavelength.

Figure 7 shows the emission spectra of $Y_4Al_2O_9:Eu^{3+}$ phosphor with different concentration of Eu . The emission spectra are characteristic $4f_6$ energy level transition emission. They are mainly due to two dipole transitions. One is 5D_0 to 7F_1 magnetic dipole transition, and the other is 5D_0 to 7F_2 forced electric dipole transition. The intensity ratio of 5D_0 to 7F_2 to 5D_0 to 7F_1 can be viewed as a clue concerning the nature

FIGURE 7: PL spectra of $Y_4Al_2O_9:Eu$ doped phosphor with variation of Eu concentrations excited with 237 nm.

FIGURE 8: PL excitation spectra of $Y_4Al_2O_9:Eu$ (1.5%) doped phosphor excited with 612 nm.

of the chemical surroundings of the luminescent center and its symmetry [24].

The strong emission peak of $Y_4Al_2O_9:Eu^{3+}$ phosphor is due to forced electric dipole transition of 3D_0 to 7F_2 centered at 611 nm. It is characteristic red emission. The $Y_4Al_2O_9$ phase crystallizes in monoclinic system with space group $P2_1/c$. Then Eu^{3+} ions with C_{2h} point symmetry are in the strict inversion center. Therefore, the phosphor should mainly exhibit the orange $^5D_0-^7F_1$ emission (for 591 nm). However, the strong emission peak of $Y_4Al_2O_9:Eu^{3+}$ crystalline phosphor is $^5D_0-^7F_1$ transition (centered at 591 nm) and $^5D_0-^7F_2$ transition (centered at 611, 624 nm). It is possible

FIGURE 9: PL spectra of $Y_4Al_2O_9:Eu$ doped phosphor with variation of Eu concentrations excited with 265 nm.

FIGURE 10: PL spectra of $Y_4Al_2O_9:Eu$ doped phosphor Eu (1.5 mol%) excited with 265 nm.

that some Eu^{3+} ions deviate from the inversion center of the crystal lattice.

The emission spectrum of phosphors was recorded by excitations with 237 nm and 265 nm. The emission spectrum is shown in Figures 7, 9, and 10 which is composed of $^5D_0-^7F_1$ ($J = 1, 2, \text{ and } 4$, as labeled in the figure) emission lines of Eu^{3+} . In general, when the Eu^{3+} ion is located at crystallographic site without inversion symmetry, its hypersensitive forced electric-dipole transition $^5D_0-^7F_2$ red emission dominates in the emission spectrum. If the Eu^{3+} site

FIGURE 11: TL glow curve of Eu^{3+} doped $Y_4Al_2O_9$ phosphor variation with Eu concentration for 5 min UV.

possesses an inversion center, $^5D_0-^7F_1$ orange emission is dominant [25]. The distinct emission lines lying between 580 and 650 nm are observed due to transitions from excited 5D_0 to the 7F_J ($J = 0-3$) levels of Eu^{3+} ions. The origin of these transitions (electric dipole or magnetic dipole) from emitting levels to terminating levels depends upon the location of Eu^{3+} ion in $Y_4Al_2O_9$ lattice and the type of transition is determined by selection rule [26]. The weak peak at 594 nm is ascribed to the magnetic dipole transition of 5D_0 and 7F_1 levels. The most intense peak emission at 612 and 628 nm corresponds to the hypersensitive transition between the 5D_0 and 7F_2 levels due to forced electric dipole transition mechanism. All the PL emission intensities for different excitations are presented in Table 1 for better understanding.

3.6. Thermoluminescence Study. Figures 11, 12, and 13 show typical TL glow curves for the Eu^{3+} doped $Y_4Al_2O_9$ phosphor exposed to various doses of UV rays from a 254UV source. The undoped sample has a well-resolved glow peak at $\sim 119^\circ C$ along with shouldered peaks at $276^\circ C$. However, in Eu doped samples, two glow peaks at 119 and $275^\circ C$ were observed. The variation of TL glow peak intensity as a function of Eu concentration was studied. It is observed that TL intensity increases linearly with Eu concentration up to 1 mol%. Thereafter, it decreases with increasing concentration of Eu. Two TL glow curves were observed at $119^\circ C$ and $276^\circ C$, respectively. The TL peak at $276^\circ C$ is well separated from other low-temperature peaks with less fading of TL signal even at elevated temperatures; TL signal is insensitive to visible light exposure. Based on these findings, $276^\circ C$ looks very attractive for TL dosimetry application. The studied samples demonstrate good repeatability of TL signal, less fading, even after repeated irradiations.

TABLE 1: PL emission intensities for different excitations.

S. number	Sample name	λ_{exe}	λ_{emi}	Intensity
1	$\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}$ (0.1%)	254	468, 594, 612, 624	46, 42, 17, 14
2	$\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}$ (0.2%)	254	468, 583, 591, 612, 624, 629	72, 55, 69, 121, 71, 62
		265	468, 583, 591, 612, 624	67, 26, 35, 53, 27
3	$\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}$ (0.5%)	265	468, 583, 591, 612, 624, 629	68, 95, 123, 247, 162, 147
		254	468, 583, 591, 612, 624, 629	74, 91, 112, 189, 125, 115
4	$\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}$ (1%)	254	468, 583, 591, 612, 624, 629	76, 90, 122, 269, 136, 124
		265	468, 583, 591, 612, 624, 629	76, 183, 260, 599, 352, 316
5	$\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}$ (1.5%)	265	468, 583, 591, 612, 624, 629	69, 158, 222, 504, 318, 277
		254	468, 583, 591, 612, 624, 629	93, 157, 204, 527, 256, 223
6	$\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}$ (2%)	254	468, 583, 591, 612, 624, 629	95, 129, 181, 436, 221, 193
		265	468, 583, 591, 612, 624	67, 26, 35, 53, 27
7	$\text{Y}_4\text{Al}_2\text{O}_9:\text{Eu}$ (2.5%)	254	468, 583, 591, 612, 624, 629	65, 141, 223, 454, 229, 213

FIGURE 12: TL glow curve of Eu^{3+} doped $\text{Y}_4\text{Al}_2\text{O}_9$ phosphor variation with Eu concentration for 10 min UV.

According to experimental results here described, the presence of transition metal ions changes the TL glow curve structure either enhancing or quenching the TL efficiency. These changes are a consequence of the crystalline field perturbation due to the different characteristics of the dopant ions which supposedly replaces the yttrium sites. The traps and the glow curve structure are also dependent upon the morphology of the surface area which in turn depends on the nanocrystallite size. The nanocrystallite size depends also on the dopant ion. Furthermore, the obtained experimental results show that the presence of dopant ions also modifies the TL recombination efficiency which was found to be different for each irradiation type and the specific exposed material. It is important to notice that using the right dopant concentration, it is possible to maximize the TL efficiency and improve sensitivity and dose linearity for a specific irradiation type.

FIGURE 13: TL glow curve of Eu^{3+} doped $\text{Y}_4\text{Al}_2\text{O}_9$ phosphor variation with Eu concentration for 15 min UV.

Thermoluminescence (TL) phosphors generally exhibit glow curves with one or more peaks when the charge carriers are released. The glow curve is characteristic of the different trap levels that lie in the band gap of the material. The traps are characterized by certain physical parameters that include trap depth (E) and frequency factor(s). For many TL applications, clear knowledge of these physical parameters is essential. In the study of relatively deep trapping effect states in various solid state materials as well as TL dating, a detailed analysis of TL glow curves is indispensable.

In this work, Chen's method [27] was used to determine the kinetic energy parameters of the glow peak of the TL materials. This method is mainly based on the temperature T_m , T_1 and T_2 , where T_m is the peak temperature, while T_1 and T_2 are temperatures at half the intensity on the ascending and descending parts of the glow peak, respectively. To determine the kinetic parameter, the following shape parameters

TABLE 2: Shape factors (μ), activation energy (E), and frequency factor(s) for UV irradiated $Y_4Al_2O_9$:Eu doped phosphor.

Eu concentration	T_1	T_m	T_2	τ	δ	ω	$\mu = \delta/\omega$	Activation energy (E)	Frequency factor(s)
0.1% Eu	64	94	124	30	30	60	0.5	0.58	1×10^9
0.2% Eu	96	127	157	31	30	61	0.49	0.66	3×10^9
1% Eu	60	87	125	27	38	65	0.58	0.63	1×10^9
1.5% Eu	87	119	161	32	42	74	0.56	0.63	2×10^9
2% Eu	96	129	168	33	39	72	0.54	0.64	2×10^9
2.5% Eu	87	117	151	30	34	64	0.53	0.66	2×10^9

are to be determined: the total half intensity width $\omega = T_2 - T_1$. The high temperature half width $\delta = T_2 - T_m$ and the low temperature half width $\tau = T_m - T_1$ and the peak shape method is mainly used to calculate the order of kinetics. Order of kinetic can be evaluated from the symmetry factor (μ_g) of the glow peak. μ_g is calculated using (2) from the known peak shape parameters δ and ω :

$$\mu_g = \frac{\delta}{\omega} = \frac{T_2 - T_m}{T_2 - T_1}. \quad (2)$$

Order of kinetics depends on the glow peak shape. The value of μ_g for the first order and second order kinetics is 0.42 and 0.52, respectively. Chen has provided a plot which gives off geometric factor (μ_g). Another parameter proposed by Balarin gives the kinetic order as a function of the parameter:

$$\gamma = \frac{\delta}{\tau} = \frac{T_2 - T_m}{T_m - T_1}. \quad (3)$$

For the first order kinetics, the Balarin parameter (γ) ranges from 0.7 to 0.8 for the second order kinetics (γ) varies from 1.05 to 1.20. Generally, in the first order, the process of retrapping is negligible and the trap should be situated very close to the luminescent center. The characteristics of the second order peak are wider and it is more symmetric than the first order peak. For a fixed heating rate, in first order kinetics, both peak temperature and shape are independent of the initial trapped electron concentration but, in the second order, the peak temperature and shape are strongly dependent on initial trapped charge concentration.

The activation energy (E) can be calculated by the general expressions formulated by Chen and is given by

$$E = C_\alpha \frac{kT_m^2}{\alpha} - b_\alpha 2kT_m, \quad (4)$$

where k is Boltzmann constant. T_m is peak temperature. The constant C_α and b_α were also calculated by Chen's equation. The activation energy and the frequency factor were seen in Table 2.

3.7. CIE Coordinate. The CIE coordinates were calculated by spectrophotometric method using the spectral energy distribution of the $Y_4Al_2O_9$:Eu³⁺ sample (Figure 14). The color coordinates for the Eu doped sample are $x = 0.566$ and $y = 0.393$ (these coordinates are very near to the orange-red light emission). Hence this phosphor has excellent color tenability from orange-red light emission.

FIGURE 14: CIE coordinates depicted on 1931 chart where $X = 0.566$ and $Y = 0.393$ (orange emission $\lambda_{\text{emission}} = 612$ nm) of Eu (1.5%) doped $Y_4Al_2O_9$ phosphor.

4. Conclusion

$Y_4Al_2O_9$:Eu³⁺ phosphor powder was successfully synthesized using a modified solid state method. XRD studies confirm that the phosphors are in single phase and nanocrystallites. $Y_4Al_2O_9$:Eu³⁺ (1.5%) phosphor shows an orange-red emission under 254 nm excitation. The photoluminescence study shows that the emission intensity of electric dipole transition (612 and 624 nm) ($^5D_0 \rightarrow ^7F_2$) dominates over that of magnetic dipole transition ($^5D_0 \rightarrow ^7F_1$) (591 nm). The optimum concentration of Eu³⁺ in $Y_4Al_2O_9$:Eu³⁺ was 1.5 mol%. The results indicated that present phosphor could find application for GaN-based UV-LED.

The $Y_4Al_2O_9$ phase was quenched in favor of the red emission of Eu³⁺ ions indicating that europium must be close to yttrium aluminate monoclinic host for better host to Eu energy transfer. However, under 254 nm excitation, Eu (1.5%) doped $Y_4Al_2O_9$ phosphor shows high intensity. The PL studies concluded that $Y_4Al_2O_9$:Eu³⁺ doped phosphor under 254 nm excitation can act as a single host for producing orange-red light with good intensity for all practical display devices in particular fluorescent lamps and CFLs. In thermoluminescence study, maximum peaks show second order kinetics which means that more than one luminescent center is present in the phosphor sample. Sample shows very good

TL glow curve. TL glow curve was analyzed and the trap depths for the two luminescence centers 119 and 276°C glow peaks were calculated. Hence this phosphor may find use in radiation dosimetry.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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