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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERISTICS OF EU3+ DOPED AL2SR2LA2O8 PHOSPHOR USING PHOTOLUMINESCENCE

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ABSTRACT

In the present study, the following samples, namely $\mathbf{Al_2Sr_2La_2O_8}$ phosphor various concentrations of Eu doped were synthesized using solid state reaction method heated at 1200^{o}C for 2 hours in muffle furnace with a heating rate of 5^o K/min. All the phosphors materials were studied for PL and few phosphors which displayed better PL emission among them were selected and studied with XRD, SEM, EDS and CIE color co-ordinates techniques were calculated. However the PL studies of the final products were found to be interesting from display device point of view.

INTRODUCTION

The luminescence associated with Eu in different host lattices has found applications related to its near UV- blue to red light emission, which is important in the field of displays. The past few decades have seen a lot of work reported on the use of divalent/trivalent Europium as a dopant in phosphors as they have very good optical properties (in the blue to red regions) which make them as a part of many display devices. Among all the rare-earth ions, Eu^{3+} is the most extensively studied, owing to the simplicity of its spectra and stability in host led to use in commercial red phosphors. Many workers reported work on La, Al, Sr oxides as host materials and Eu^{3+} as dopant [1-6]. When the phosphor is prepared in reducing atmosphere getting the final phosphor is mostly in Eu^{2+} state however few percentages in Eu^{3+} also found [7-8]. Few display phosphors thermoluminescence also studied [10-12].

Experimental

Aluminum oxide, Strontium carbonate, Lanthanum oxide were taken as raw materials for the host and the molar ratio of rare earth Europium oxide taken as activator ion which is doped in host at different concentrations. All the chemicals were purchased from National Chemicals, Baroda, of assay 99.9%. The base materials and activator were mixed and ground thoroughly using agate mortar and pestle prior to this all the materials are weighed as per the required quantities. Acetone is added to get uniform mixing of the oxides while grinding using agate mortar and pestle for 30 minutes. $Al_2Sr_2La_2O_8$ Phosphor is synthesized using standard solid state reaction with varying Eu (0.1, 0.2, 0.3, 0.4, 0.7, 1.0, 1.5, 2.0 and 2.5%) molar concentration. To prepare Aluminum Strontium Lanthanate (ASL) doped with various concentrations of Eu, consists of heating stoichiometric amounts of reactants at 1200°C for 2

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hours in a muffle furnace. The phosphor materials were cooled to room temperature naturally. The received powder being ground thoroughly using an agate mortar, to ensure the best homogeneity and reactivity.

The following is the final basic reaction used to prepare the ASL:Eu$_x$ phosphors

$$Al_2O_3 + La_2O_3 + 2SrCO_3 + Eu_2O_3 \xrightarrow[\text{Air}]{1200\,^{\circ}C,\ 2\ Hrs} Al_2Sr_2La_2O_8{:}\ Eu$$

RESULTS AND DISCUSSIONS

Photoluminescence study

Fig:1 is the Excitation & Emission spectra of Eu(1.5%) doped $Al_2Sr_2La_2O_8$ phosphor when excited with 254,268 and 278 nm. When the Eu is 0.3% or more in ASL phosphor the entire excitation spectra shifted from 254nm to band of 250-340nm peaking at 268, 278nm while monitoring at 627nm. ASL excitation peak intensity increases up to 1.5% of Eu in ASL and then decreases as Eu concentration increases in ASL.

The highest intensity is for Eu 1.5% in ASL around 800 units with broad absorption 250 to 340 nm, which give us to design a display device for wide range of excitation, which is 100 nm band in 250-350nm. However the present phosphor deals only with 254 nm (mercury resonance radiation) commonly used and easily producible low pressure mercury discharge. This low pressure mercury discharge is used in fluorescent and compact fluorescent lamps all over the world. When the Eu doped ASL phosphor is excited with 254nm, 268, 278 nm the following emission peaks with different intensities are observed: 468,514,535, 556,588,615, 627 nm peaks apart from less intensity close emissions with different intensities.

However the 541, 588 nm peak intensities are same and 627nm peak intensity is more by 20% on comparing with 541 and 588nm.

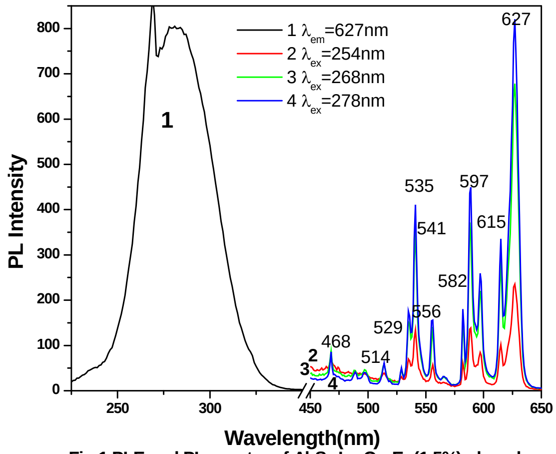


Fig 1 PLE and PL spectra of $Al_2Sr_2La_2O_8$: Eu(1.5%) phosphor

It is also interesting to note that as Eu concentration varies, the main peaks 541 & 588nm peaks vary their emission intensities as well as wavelengths. The peak at 627 nm (red) grows its intensity without affecting the wavelength, when Eu concentration increased in ASL. From fig. it is found as excitation of Eu increases, the prominent peak intensities increase by 4 times along with all other sibling peaks. This is due to stranded Eu-O, Sr-Eu-O, resonance absorption by the ASL:Eu phosphor.

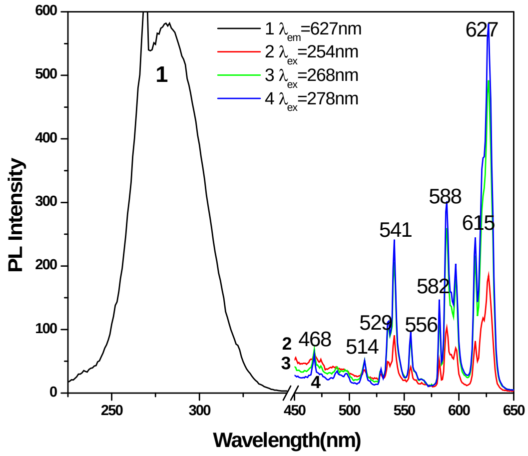


Fig 2 PLE and PL spectra of $Al_2Sr_2La_2O_8$: Eu(2.5%) phosphor

Fig 3 is the PL emission spectra of ASL phosphor excited with 268 nm for all Eu concentrations in ASL phosphor. Fig 4 is the behavior of PL emission of ASL for different concentrations of Eu.

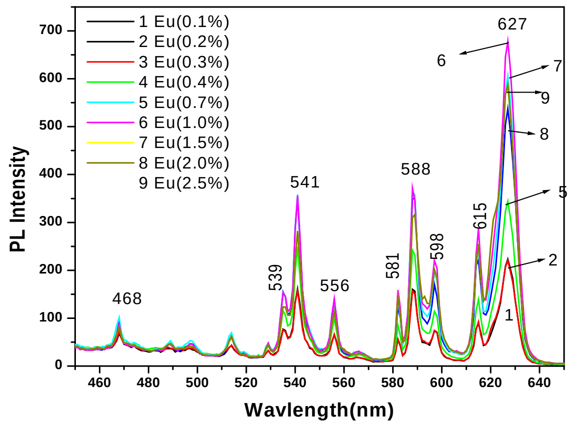


Fig.3 PL spectra of Eu concentrations of under 268nm Excitation

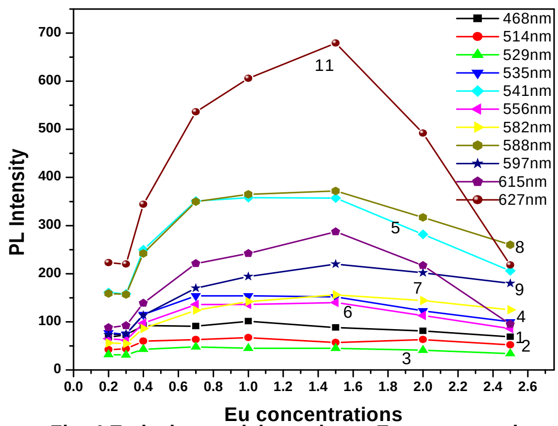


Fig. 4 Emission peak intensity vs Eu concentrations under 268nm excitation

From the figure it is found that, the intensities of 535, 588 and 627 nm peaks increase their intensities upto 1.5% Eu in ASL phosphor and decrease their intensity marginally in ASL :Eu (0.1%, 0.2 %, 0.3%, 0.4% , 0.5%, o.7%, 1.0%, 1.5% , 2%, 2.5%.).

Table 1 shows the intensities of various emission peaks and wavelength of ASL: Eu (0.1%, 0.2 %, 0.3%, 0.4%, 0.5%, 0.7%, 1.0%, 1.5%, 2%, 2.5%.) phosphor under 268 nm excitation. Table 1 and Figure 4 are presented for easy understanding and better comparison of PL emission found in ASL : Eu (0.1%, 0.2 %, 0.3%, 0.4% , 0.5%, o.7%, 1.0%, 1.5% , 2%, 2.5%.). It is also noted that all the PL emission are allowed transitions of Eu^{3+} in figure 5.

XRD study

XRD study reviles various crystallite sizes and phase identifications. The strong and sharp peaks indicate that highly crystallized and structurally ordered at long-range. The average crystallite size had been estimated by the Scherrer's equation using the full width at half maximum (FWHM) for the intense peak (1 0 3).

The average crystallite size was calculated using the Debye-Scherrer formula given in the literature, i.e., $d = K\lambda/\beta\cos\theta$. Where k is constant (0.9), λ is the wavelength of the x-rays used (0.154 nm in the present case), β is the full width at half maxima (FWHM), θ is the Bragg angle of the XRD peak.

Table 1 Intensity of various emission peaks of $Al_2Sr_2La_2O_8$:Eu phosphors under 268nm excitation

S.No.	Excitation Wavelength	Eu Concentration (mol%)	Different Emission peaks under 268nm Excitation (nm)										
			468	514	529	535	541	556	582	588	597	615	627
1		0.1	41	23	17	37	73	29	24	70	32	38	91
2		0.2	68	42	32	77	161	65	56	159	73	88	223
3		0.3	73	44	31	74	158	62	55	157	75	92	220
4		0.4	92	60	43	115	250	97	86	242	114	139	344
5	268nm	0.7	91	63	48	154	351	136	124	350	170	221	536
6		1.0	101	67	45	154	358	136	142	365	194	242	606
7		1.5	88	57	45	152	357	140	156	372	220	287	**679**
8		2.0	81	63	41	123	282	113	144	317	202	217	492
9		2.5	69	52	34	100	206	86	125	260	180	95	218

The 627 nm peak which is red emission from Eu^{3+} state is interesting to observe. As Eu concentration increases, the emission intensity of 627nm from Eu^{3+} ion increases linearly. The emission at 541 nm is due to $^5D_1 \rightarrow {^7F_1}$ transition. The emissions at 588 and 627nm are due to $^5D_0 \rightarrow {^7F_1}$ and $^5D_0 \rightarrow {^7F_2}$. All these emissions in ASL:Eu phosphors are the allowed transitions of Eu^{3+}, which are due to electric dipole moment and magnetic dipole moment of crystal lattice. The emission at 365nm is due to the crystal field of the phosphor, which was reported by many workers. The emission around 469 nm may be due to Al – Sr – O bonding in the ASL: Eu phosphor material.

Table 2

Sr.No	Peak Wavelength (nm)	Intensities (a.u) Without flux under 254nm excitation					
		Eu (0.1%)	Eu (0.2%)	Eu (0.5%)	Eu (1%)	Eu (1.5%)	Eu (2%)
	468 $^5D_2 \rightarrow {^7F_0}$	68	78	80	85	80	77
	541 $^5D_1 \rightarrow {^7F_1}$	53	82	140	171	156	137
	588 $^5D_0 \rightarrow {^7F_1}$	43	76	149	177	182	173
	627 $^5D_0 \rightarrow {^7F_3}$	50	87	198	264	281	291

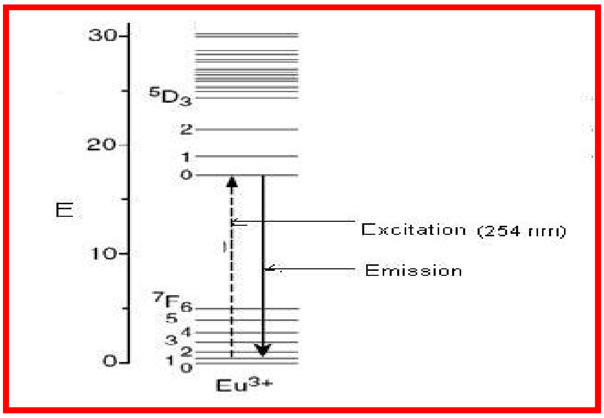


Figure 5 Energy Level Diagram of Eu^{3+}

From the XRD it is found most of the phosphors are in single phase.

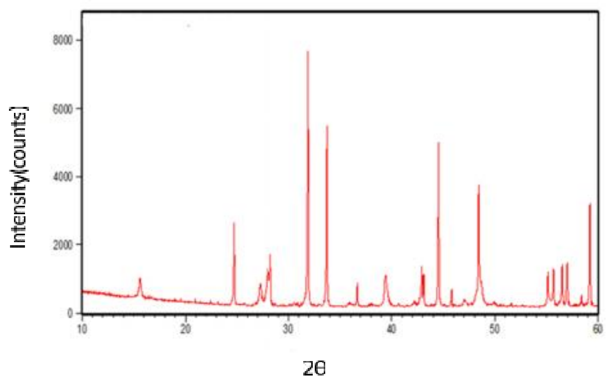
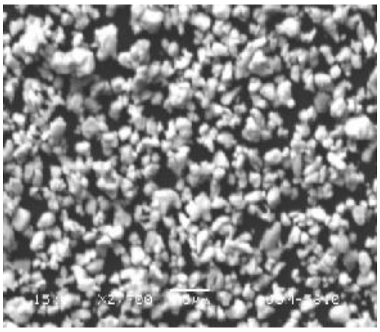


Fig.6A XRD pattern of $Al_2Sr_2La_2O_8$: Eu (1.5%) phosphor

It is found that for ASL: Eu (1.5%) phosphor, the crystallite size is 55.10nm. As the concentration of Eu increases to 2.5% in ASL, the crystallite size increases to 59nm.

SEM study

From the SEM studies the particle size variations and the agglomerations are seen for phosphors.



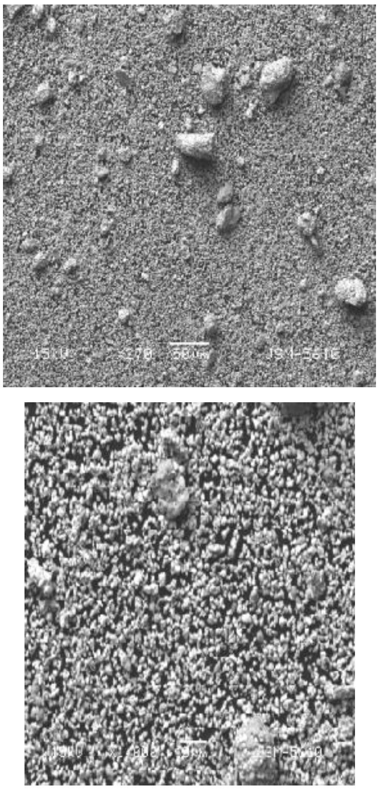


Fig.7A SEM images of 1.5% Eu doped $Al_2Sr_2La_2O_8$ phosphor

Fig.7A is the SEM micrographs of ASL: Eu (1.5%), phosphor of 550, 1000 and 2700 magnification. The 2700 magnification is of 5 microns scale, wherein the particles are nearly spherical in shape having size 2 – 4 microns. This phosphor exhibited highest PL intensity as shown in Fig.1.

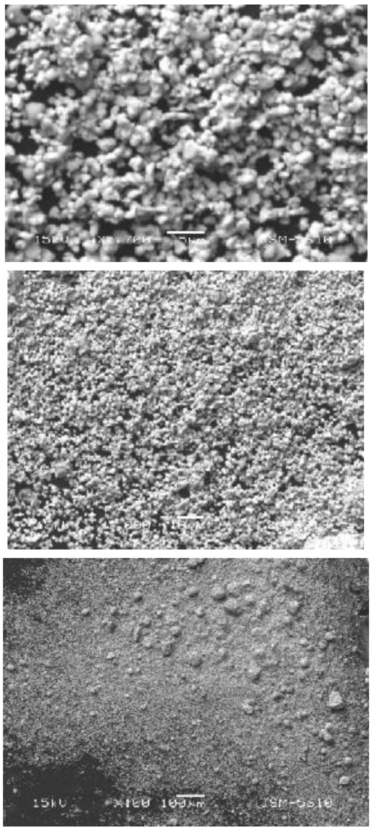


Fig.7B SEM images of 2.5% Eu doped $Al_2Sr_2La_2O_8$ phosphor

Fig.7B is the SEM micrographs of ASL: Eu (2.5%), phosphor of 550, 1000 and 2700 magnification. The 2700 magnification is of 5 microns scale, wherein the particles nearly spherical in shape having size 2 – 4 microns. This phosphor exhibited reduced PL intensity in comparison with Figures 1 and 2.

EDS study

From the EDS spectrum, it is found most of the phosphors consist of host and dopants only

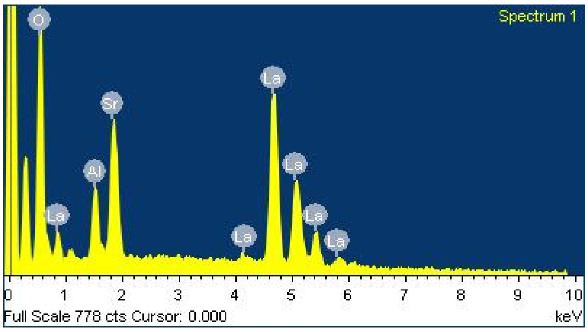


Fig. 8 EDS spectrum of 2.5% Eu doped $Al_2Sr_2La_2O_8$ phosphor

However Figure 8 show the presence of few impurity elements like Nickel, Cobalt. Other elements are in ppm level, which can be found from intensities on the Y – axis of EDS (Energy Dispersion Spectrum) spectra of different phosphors. The impurities Nickel and Cobalt come from the rare earth oxides used in the preparation and are present in trace level.

CIE analysis

Fig. 9 shows the CIE co-ordinates of ASL: Eu(2%) phosphor prepared without flux. The CIE co-ordinates of (chart-1931) were calculated by the Spectrophotometric method using the spectral energy distribution of ASL: Eu(2%) phosphor prepared without flux. The colour co-ordinates found from the diagram are A. x=0.470, y=0.480, which indicate that the phosphor without flux emission colour is in the orange region.

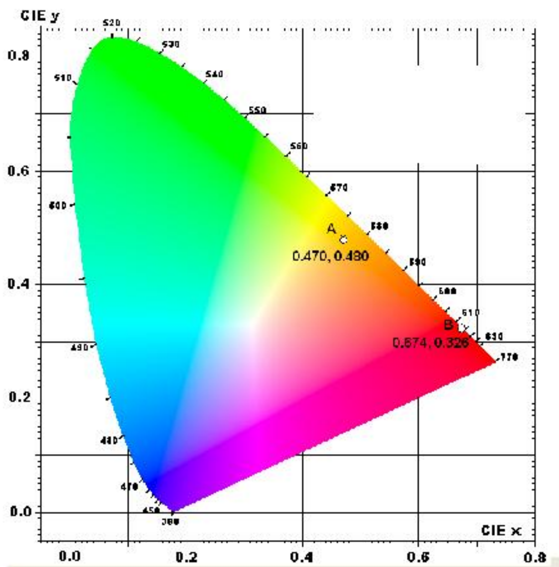


Fig.9 CIE co-ordinates of ASL:Eu (2%) phosphor A) without flux

CONCLUSIONS

- All the peaks observed at 468, 541, 588, 598 and 627 are the allowed transitions of Eu^{3+}; the intensities of all the observed peaks are relatively good for ASL:Eu prepared without flux
- It is concluded that ASL: Eu prepared using solid state reaction gives all the primary colours: blue, green, yellow and red, which is the basic requirement for the generation of white light in a display device. However the present phosphor ASL:Eu(1.5%) is red dominated phosphor.
- XRD analysis shows that the synthesized composition retains the mostly single phase. For ASL: Eu (1.5%) phosphor, the crystallite size is 55.10nm.
- SEM investigation confirms the particles nearly spherical in shape having size 2 – 4 microns.
- EDS technique the impurities Nickel and Cobalt come from the rare earth oxides used in the preparation and are present in trace level.
- The colour co-ordinates found from the diagram are A. x=0.470, y=0.480, which indicate that the phosphor without flux emission colour is in the orange region.

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