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Synthesis and Characterization of Dy^{3+} Doped ASL: Eu^{3+} Phosphor

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Abstract— *$AlSrLaO_4$ phosphors were synthesized using standard solid state reaction [SSR] method with varying Dy molar concentration as Dy (0.1, 0.2, 0.3, 0.4, 0.5, 0.7, 1.0, 1.5, 2.0 mol %) at Eu(0.5%) constant. The mixture of reagents was ground together to obtain a homogeneous powder in acetone base. To prepare Aluminum Strontium Lanthanate (ASL) doped with various concentrations of Dy, consists of heating stoichiometric amounts of reactants at 1000°C for 2 h in a muffle furnace. The received powder being ground thoroughly using an agate mortar, to ensure the best homogeneity and reactivity, powder was transferred to alumina crucible, and then reheated in a muffle furnace at 1200°C for 4 hours. The phosphor materials were cooled to room temperature naturally. All samples were found out to be white who are studied for photoluminescence [PL]. Photoluminescence spectra were recorded at room temperature using Shimadzu-5301 Spectrofluorophotometer. The phosphors are characterized using XRD, SEM,EDS and Particle size histogram. Dy shows all its primary allowed emissions hence it is concluded that this phosphor can be used in display devices.*

Keywords— *Photoluminescence, X-ray diffraction [XRD], Scanning Electron Microscopy [SEM], Energy Dispersion Spectrum [EDS], $AlSrLaO_4$ [ASL], Solid State Reaction [SSR].*

1. 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].

2. 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. $AlSrLaO_4$ 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 1000 °C for 2 hours in a muffle furnace. The received powder being ground thoroughly using an agate mortar, to ensure the best homogeneity and reactivity, powder was transferred to alumina crucible, and then reheated in a muffle furnace at 1200 °C for 4 hours. The phosphor materials were cooled to room temperature naturally.

The following is the final basic reaction used to prepare the ASL:Eu_x phosphors

$$Al_2O_3 + La_2O_3 + 2SrCO_3 + Eu_2O_3 + Dy_2O_3 \xrightarrow[\text{Air}]{1200\ ^oC,\ 2\ Hrs} 2AlSrLaO_4\text{: Eu, Dy}$$

3. RESULTS AND DISCUSSIONS

3.1 Photoluminescence Study

Fig. 1 is the Excitation & Emission spectra of Eu(0.5%), Dy(0.3%) doped $AlSrLaO_4$ phosphor. The following

emissions with different intensities are found when excited with 254 and 268nm are 468, 514, 541, 556, 588, 597, 615 and 627nm. Among the observed peaks 627, 588 and 541 are predominating. Among these 627nm peak is highest in intensity. Apart from these there are another four peaks in green-red region as satellite peaks. All the emissions are allowed transitions of Eu^{3+} and Dy^{3+}.

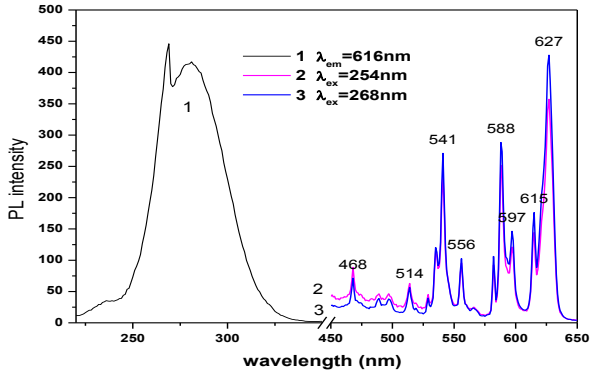

Fig. 1: Ex. & Em. spectra of AlSrLaO4; Eu(0.5%), Dy(0.3%) phosphor

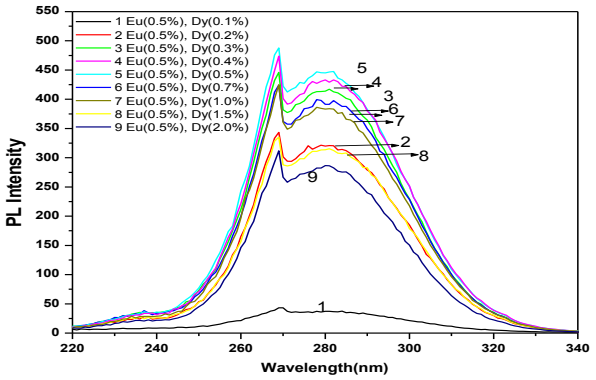

Fig. 2: Excitation peaks of $AlSrLaO_4$

Fig: 2 is the combined Excitation spectra of $AlSrLaO_4$: Eu for different Dy concentrations. From the figure the maximum absorption occurs for 0.5% of Dy in $AlSrLaO_4$.

Fig: 3 is the combined emission spectra of $AlSrLaO_4$: Eu for different Dy concentrations. When excited with 254nm the intensity and emitted wavelength for various Dy concentration in $AlSrLaO_4$:Eu when excited with 254nm are presented in table 1. From the table it is found the main emission peak at 627nm grows its intensity up to Dy(0.5%) in $AlSrLaO_4$:Eu.

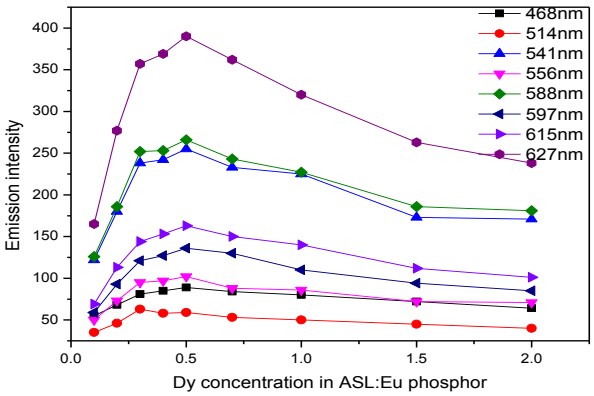

Fig. 3: Emission peak intensities vs Dy concentrations in ASL:Eu phosphor under 254nm ex.

3.2 XRD Study

XRD study (Fig. 4) 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., $d = k/$, 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. From the XRD it is found most of the phosphors are in single phase. The crystallite size is calculated using Scherer's formula for all the samples using $D = K\lambda/\beta\cos\theta$.

Table 1: Intensity of various emission peaks of Dy (0.1, 0.2, 0.3, 0.4, 0.5, 0.7, 1.0, 1.5, 2.0 mol %) of AlSrLaO4:Eu^{3+} phosphor under 254nm excitation

S.No	Sample	Emission intensities under 254nm							
		468	514	541	556	588	597	615	627
1	$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(0.1%)	55	35	122	50	126	59	69	165
2	$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(0.2%)	68	46	180	73	186	93	113	277
3	$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(0.3%)	81	63	238	95	252	121	144	357
4	$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(0.4%)	85	58	242	97	253	127	153	369
5	$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(0.5%)	89	59	255	102	266	136	163	390
6	$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(0.7%)	84	53	227	88	229	113	137	332
7	$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(1.0%)	75	51	220	86	227	109	138	327
8	$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(1.5%)	72	45	173	72	186	94	112	263
9	$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(2.0%)	64	40	171	71	181	85	101	238

From the fig.4 it is found irrespective of dopant concentration (Eu, Dy) in ASL phosphor the crystallite size is 55.10nm.

3.3 SEM Study

From the SEM studies (Fig. 5) the particle size variations and the agglomerations are seen for phosphors under study.Figure 5 is the SEM micrographs of ASL: Eu (0.5%), Dy (0.3%) phosphor of 550, 1000 and 2700 magnification. The 2700 magnification is of 5 microns scale where in the particles nearly spherical in shape having size 2 – 6 microns.

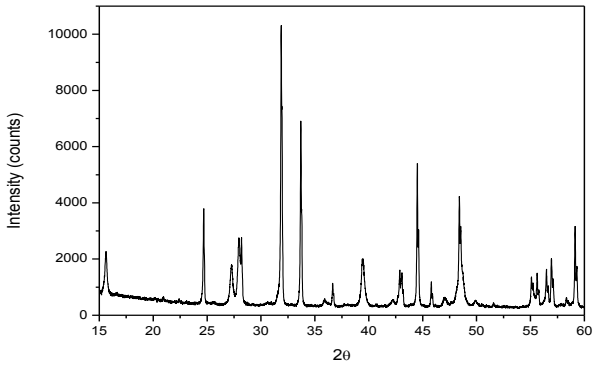

Fig. 4: XRD pattern of 0.3% Dy doped ASL:Eu(0.5%) phosphor

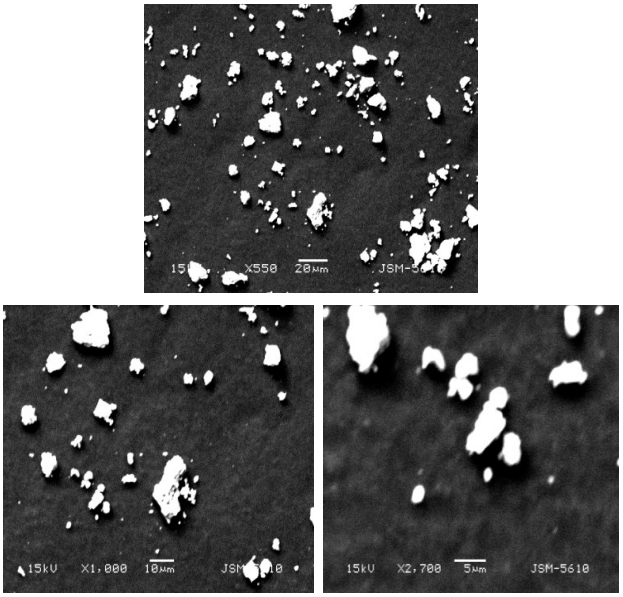

Fig. 5: SEM images of 0.3% Dy doped ASL:Eu (0.5%) phosphor

3.4 EDS Study

Fig. 6 is the EDS spectrums of the phosphor under characterization. From the EDS spectrum it is found most of the phosphors consist of host and dopants only. However it consists of few impurity elements like Nickel, Cobalt, Gallium, Tantalum, Tungsten, and Copper. The Cu & W may be from the instrument accessories. 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, Cobalt, Gallium, and Tantalum are from the rare earth oxides.

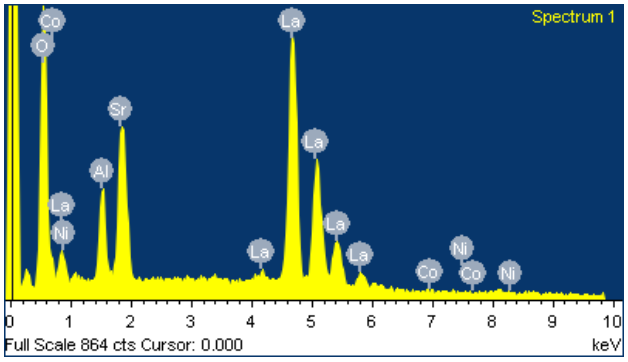

Fig. 6: EDS spectrum of 0.3% Dy doped ASL:Eu(0.5%) phosphor

3.5 Particle Size Analysis

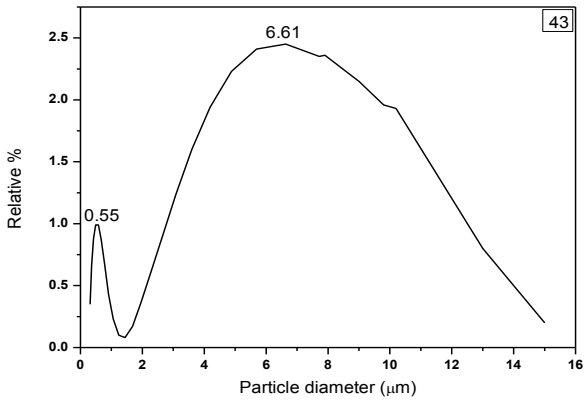

Fig. 7: Particle size histogram of 0.3% Dy doped ASL: Eu (0.5%) phosphor

Table 2

Sr. No	*Sample Name*	*Concentration (mol%)*	*Particle size (microns)*	*Specific Surface area (m^2/gram)*
1	$AlSrLaO_4$	Eu(0.5%), Dy(0.3%)	0.55, 6.6	1.8011

4. CONCLUSIONS

$Al_2Sr_2La_2O_8$: Eu(0.5%), Dy(X) phosphors are synthesized successfully using SSR. $AlSrLaO_4$: Eu(0.5%), Dy(0.5%) is best as it is evident from the Table.1 all the primary emissions blue, green and red are emitted. However the red dominated white emission is observed. The particle size is 6.6 microns and the surface is 1.8018 M^2/gram. From XRD study (Eu, Dy) doped ASL phosphor the crystallite size is 55.10nm.

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