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Characterization and Optical Properties of Eu and Dy Doped SrAl₂O₄ Phosphors*

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Abstract: In this paper we report the synthesis, characterization and optical properties of $SrAl_2O_4$ phosphors prepared by combustion technique. The samples are prepared at 600°C for 5 minutes. The as prepared samples are characterized through SEM and XRD measurements. The XRD pattern exhibits multiple peaks corresponding to $SrAl_2O_4$ while SEM micrographs show non-uniform distribution of grains. The average particle size of the sample is estimated to lay around 120 nm. The Photoluminescent (PL) emission spectrum peaks at 446 nm, which is attributed to $4f^65d^1$ to $4f^7$ configuration of Eu ions.

Key words: Optical properties, combustion technique, SEM, XRD, photoluminescence.

1. Introduction

In last decade nano sized materials have drawn the attention of scientists and researchers worldwide because of their potential impact in many fields like photonics, electronics, sensing and catalysis¹. These materials possess large number of applications in the areas of organic solar cells, carbon nano tubes (CNTs), solid state batteries, super plastic ceramics, multifunctional materials, molecular electronics, biosensors and lasers². Such materials take advantage of size induced changes in structural, optical and electronic properties to create enhanced luminescent materials, whose properties differ

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from the corresponding bulk phase³. Phosphors are one of those materials which offer promising applications when prepared in nano order. Aluminates of alkaline earth doped with europium (Eu) and dysprosium (Dy) form phosphors which are efficient and long lasting with high quantum efficiency⁴. These phosphors are widely accepted because of their good luminescent properties such as high initial intensity, long decay time, chemical stability, suitable emission color and no radiation. These oxide phosphors exhibit a long period of luminescence and the lasting time of this new kind of phosphors is more than 10 times then that of earlier used sulphides⁵.

Many researchers all over the world have been trying to synthesize these phosphors for display applications and long life luminescence. Several kinds of chemical synthesis techniques such as sol-gel⁵, reverse micro emulsion⁶, co-precipitation⁷ and combustion methods⁸ have been applied to prepare SrAl₂O₄ and its phosphors. Among these, combustion process is an interesting method to prepare the precursor powders⁹. This technique is based on the use of heat energy released by the redox reaction between the metal nitrate and urea or other fuels inflammable at relatively low temperature. This method is safe as well as energy saving. This paper investigates and reports preparation of SrAl₂O₄ phosphor doped with Eu²⁺ and Dy³⁺ by using combustion technique.

2. Experimental Techniques

2.1. Sample Preparation

The materials used for synthesis are strontium nitrate, aluminium nitrate and urea (All AR grade 99.9% pure). Apart from this oxides of Eu and Dy are taken as dopants. The composition used in the present study is $Sr_{0.97}$ $Eu_{0.01}Dy_{0.02}$. All these materials are taken in a alumina crucible with comparatively larger volume. The distilled ionized water in the appropriate amount is added to above materials that convert it into a paste. The alumina crucible is placed in furnace maintained at temperature around 600°C. Within 5 minutes, the furnace reaches the desired temperature and reaction starts giving yellowish flame. This continues for next few seconds and as it is over, crucible is pulled out of furnace and kept in open to allow cooling. Upon cooling we get fluffy form of material, which is then grinded to get material in powder form.

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2.2. Measuring Instruments

The samples are prepared in a digital furnace maintained a temperature of 600 $^{\circ}$ C. The XRD studies are performed using Rigaku Miniflex II using Cu-K α radiation where X-rays are generated at 30 KV/15 mA voltage and current values respectively. The morphological structures of as prepared sample are observed using JEOLJSM-6380 scanning electron microscope (SEM). For observing excitation and emission spectra Hitachi F-2500 Fluorescence spectrophotometer is used in the wavelength range 100-700 nm.

3. Results and Discussion

3.1. Characterization:

In order to ascertain the crystalline nature of the prepared samples, SEM and XRD studies are performed. The microstructures of Eu and Dy doped $SrAl_2O_4$ samples are studied by SEM.



Fig. 1 SEM micrographs of SrAl₂O₄: Eu, Dy phosphor

Fig. 1 shows SEM micrographs of strontium aluminate phosphor prepared by combustion method in which we find platelet like structure with some unoccupied space. It shows that particles are in form of clusters of irregular shape. Each smaller cluster sizes around 10 μ m with mean particle size~120 nm, calculated from Heyne's intercept method¹⁰. The lots of vacant space may be due to escape of gases during combustion reaction. In fact, the large amount of escaping gases dissipates heat and thereby prevents the material from sintering and thus provides conditions for formation of nano crystalline phase⁴.



Fig. 2 X-ray diffraction pattern of SrAl₂O₄: Eu, Dy phosphor

3.2 Optical Studies:

In order to determine the crystal structure and to establish chemical nature of the combustible product, XRD study is carried out. $SrAl_2O_4$ has two phases, a high-temperature hexagonal phase (β -phase) and a low temperature monoclinic phase (α -phase). A typical XRD pattern of the resultant $SrAl_2O_4$: Eu, Dy sample is presented in fig 2. It is clear from figure that $SrAl_2O_4$ crystal is a monoclinic lattice. As reported by earlier workers ⁴ and as seen here $SrAl_2O_4$ crystal is monoclinic lattice. The average grain size estimated from Sherrer's formula is found to lie around 125 nm ¹¹. The XRD peaks obtained corresponding to different planes are (011), (-211), (220), (211) and (031).

The spectral energy distribution of $SrAl_2O_4$ at room temperature (RT) and an excitation wavelength of 365 nm is shown in fig. 3. Sample is characterized by broad band emission spectrum centered at 446 nm along with two humps at 365 and 534 nm respectively. Probably this is corresponding to $4f^65d^1$ to $4f^7$ transition of Eu⁺² ions. Although 4f electrons are not sensitive to the lattice environment due to the shielding effect of the electrons in the inner shell, the 5d electron may couple strongly to the lattice. As a result, the mixed states of $4f^65d$ split by the crystal field and couple strongly to the lattice phonons ¹². It is already reported that the emission maxima of the

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phosphor prepared by combustion method shifts to shorter wavelength (520-516 nm)⁸. This may be attributed to changes of the crystal lattice around Eu^{+2} and quantum size effect of phosphor nano particles. Since the excited $4f^{6}5d^{1}$ configurations of Eu^{+2} ion are extremely sensitive to the change in lattice environment, the 5d electrons may couple strongly to the lattice ¹³. Hence the mixed states of 4f and 5d configurations will be splitted by the crystal, which may lead to the blue shift of its emission peak.



Fig. 3 PL emission spectra of SrAl₂O₄ phosphor

4. Conclusions

The combustion method is a reliable method to prepare $SrAl_2O_4$ phosphor. Results of XRD show the semi crystalline nature of the prepared samples while SEM exhibits non-uniform distribution of grains. The average grain size calculated from XRD and SEM observations are obtained to be around ~120 nm. The emission spectrum centered at 446 nm is associated with two secondary peaks.

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