SYNTHESIS AND OPTICAL PROPERTIES OF MN AND DY DOPED STRONTIUM ALUMINATE NANOPHOSPHORS

V.T.Jisha¹, Sajikumar.A.C²

Abstract: Luminescence investigations of Mn and Dy doped Strontium aluminate phosphors prepared through sol gel method. The prepared phosphors has been characterized by Powder X-ray diffraction, Scanning electron microscope, Optical properties were studied by photoluminescence analysis. Photoluminescence studies of the prepared phosphors showed prominent IR emission, green emission and UV emission.

INTRODUCTION
Rare-earth and non-rareearthdoped inorganic phosphors are widely used in a variety of applications, such as lamp industry, X-ray imaging, and colour display. Phosphors based on oxide matrices are attractive host materials for the development of advanced phosphors due to their ease of synthesis and stability. Rare earth doped aluminates serve as an important class of phosphors for fluorescent lamp and phosphorescence applications. Among the group Strontium aluminates are the most widely used members of the alkaline earth aluminates MAl₇O₁₈ (M = Ba, Ca or Sr) [1, 3]. They are commonly used as cements in a wide range of applications. Rare earth doped SrAl₇O₁₈ shows interesting luminescence properties [6–14] More specifically, SrAl₇O₁₈:Eu is one of the few materials exhibiting a long-lasting afterglow after excitation has ended, a phenomenon known as persistent luminescence [9,10,13]. Different kinds of techniques have been used to prepare SrAl₇O₁₈ such as solid state reaction, co-precipitation, microwave, and combustion and sol-gel synthesis. Comparing these methods, sol-gel synthesis possesses some benefits, namely, relatively low preparation temperature, easy control of the stoichiometry, high levels of product homogeneity, and no need for the use of expensive equipment. In this article, we reported the synthesis of nanostructured SrAl₇O₁₈ and Mn and Dy doped SrAl₇O₁₈ via Sol Gel synthesis.

EXPERIMENTAL
Strontium aluminates phosphors were prepared by sol-gel synthesis method. All the reagents used in the experiments were in analytical grade and used without any further purification. Effects of the Mn and Dy on PL properties were investigated. The starting materials were Strontium acetate, Aluminum acetate and, Dysprosium nitrate, and 2methoxy ethanol as a solvent. 99% of 2M Strontium acetate [(CH₃.COO)₂Ca.2H₂O] with 1% of Aluminum acetate [C₄H₆AlO₄.4H₂O] and 1% of Manganese Nitrate for SrAl₇O₁₈:Mn and 1% of Dysprosium Nitrate for SrAl₇O₁₈:Dy were used as the precursors, the acetic acid and ethylene glycol were added 1:1 ratio to the precursor solution. The acetic acid used as the gelling agent and ethylene glycol act as reaction medium. The mixture was stirred 30 minutes magnetically at room temperature. The pH of the resulting solution was adjusted at 10.5 using ammonium hydroxide. After completion of the reaction, the solution was kept under constant stirring at 80°C temperature using magnetic stirrer. After 2 hours white color viscous sols was occurred. The resultant sol gels were dried for 5 hours at 100°C. The obtained powder was annealed at 950°C for 2 hours to obtain SrAl₇O₁₈ nanopowders. The phase composition and phase structure were characterized by X-ray diffraction (XRD) pattern using Xpert PRO diffractometer with Cu Kα radiation (λ = 1.5406 Å) operating at 45kV, 40mA. The morphology and the composition of the products were examined by scanning electron microscopy (SEM, JSM-6390) equipped with an energy-dispersive spectrometry (EDS).

RESULTS AND DISCUSSION
Formation of these phosphors has been characterized by powder XRD. Fig.1(a–c) shows the representative powder XRD pattern for the SrAl₇O₁₈, SrAl₇O₁₈: Mn and SrAl₇O₁₈:Dy. It is reported that besides SrAl₇O₁₈ it was found that a pure monoclinic phase of parent SrAl₇O₁₈ is dominant in the XRD pattern (JCPDS.25-1289). The results proved that all phosphor samples prepared in this work are almost single SrAl₇O₁₈ phase, and the little amount of co-doped rare

¹ Research Centre, S.T. Hindu College, Nagercoil -629 002, Tamilnadu, India.
² Malankara catholic college, kaliyakavilai, kk.district.
earth ions have almost no effect on the SrAl₂O₇ phase composition. The small amount of doped rare earth ions has virtually no effect on phase structures. The sharp peaks indicate that the SrAl₂O₇ nanoparticles (NP) were well crystallized.

Fig.1(a-c): XRD patterns of (a) SrAl₂O₇ (b) SrAl₂O₇: Mn (c) SrAl₂O₇:Dy

The analysis of the crystallite size has been carried out using the broadening of the XRD peaks. Peak broadening comes from several sources, i.e. instrumental effect, finite crystallite size, and strain effect within the crystal lattice. Taking care of all the sources, crystallite size has been calculated using Williamson-Hall plot. Williamson and Hall plot is a classical method to obtain qualitative information of anisotropy in broadening. Williamson and Hall assumed that both size and strain. According to the Williamson-Hall method, the individual contributions to the broadening of reflections can be expressed as

\[ \beta_{hkl} \cos \theta_{hkl} = [K\lambda/D] + [4\varepsilon \sin \theta_{hkl}] \]

Fig.2(a-c): Williamson-Hall plots for (a) SrAl₂O₇ (b) SrAl₂O₇: Mn (c) SrAl₂O₇:Dy

A plot is drawn with 4sinθ along the x-axis and βcosθ along the y-axis for as prepared SrAl₂O₇ and SrAl₂O₇ : Mn, Dy nanoparticles as shown in Figure 2(a-c). From the linear fit to the data, the crystalline size was estimated from the y-intercept, and the strain ε, from the slope of the fit. Quantitative data obtained from the Williamson-Hall plot indicated that the average particle size attained for SrAl₂O₇, SrAl₂O₇: Mn and SrAl₂O₇: Dy was 72.5nm 55nm and 120nm respectively.

Fig.3(a-c) SEM images of (a) SrAl₂O₇ (b) SrAl₂O₇: Mn (c) SrAl₂O₇:Dy
The surface morphology of material in the form of granular structure with round morphology and agglomeration were investigated with a scanning electron microscope. Fig.3 (a-c) shows SEM of SrAl₄O₇, and Mn, Dy doped SrAl₄O₇. It shows uniform distribution of pores with clusters of crystallites over the entire material. The Mn and Dy doping significantly change the appearance of the particles and morphology.

![Fig.4(a-c) Emission spectra of (a) SrAl₄O₇ (b) SrAl₄O₇: Mn (c) SrAl₄O₇:Dy](image)

The photoluminescence spectra of SrAl₄O₇, Mn doped SrAl₄O₇ and Dy doped SrAl₄O₇ nanoparticles under 360 nm excitation wavelength is shown in Fig.4 (a-c) respectively. The PL emission spectra of all samples exhibit three emission bands with corresponding peak wavelengths of 395 nm for exciton emission, 520 nm for green emission attributed to oxygen interstitial and 790 nm for excitation of 360nm. SrAl₄O₇: Mn nanocrystals are found to have increased photoluminescence efficiency associated with the magnetic impurity Mn²⁺. For small particles like the SrAl₄O₇: Mn nanocrystals, majority of the Mn²⁺ ions are at the near-surface sites and occupy axial or lower symmetry sites.

CONCLUSIONS
SrAl₄O₇, SrAl₄O₇:Mn and Dy phosphor has been synthesized by the Sol Gel method. XRD patterns show the phase formation of pure SrAl₄O₇ phases. XRD analysis shows that the prepared compositions retain the monoclinic phase of SrAl₄O₇. The SEM images confirm the irregular particle shape that was produced from the sol gel method. The main peaks of emission spectra of the luminescent nanoparticles are at 395 nm, 520 nm and 790 nm. This fundamental work might be important in developing new IR imaging devices.

REFERENCES


