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PHOTOLUMINESCENCE PROPERTIES OF DY^{3+} DOPED SRS PHOSPHOR BY E- SOLID STATE DIFFUSION METHOD

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ABSTRACT

The Alkaline earth sulfide phosphors doped with Dy^{3+} or Ce3+ are good candidates for wavelength conversion in LEDs for general lighting applications. XRD, FESEM, HRTEM, SAED and EDX characterized the samples. The percentage of doping material is confirmed from the EDX spectra. The average crystal size of the prepared SrSnanopowder is determined by XRD.Photoluminescence (PL) spectra of SrS phosphors doped with trivalent Dy^{3+} ions were studied. PL emission for SrS: Dy^{3+} shows a peak at 481 nm, 580 nm and 675 nm at an excitation wavelength of 331 nm, which is yellow bands in Dy^{3+} ions show that phosphor may be applicable for white light emitting diodes LEDs.

Keyword: Luminescence, Nanoparticles,Photoluminescence

White light emitting diodes (LEDs), with their characteristics of high brightness, reliability, long life time, low environmental impact and energy efficiency [1,2] are expected to replace conventional incandescent and fluorescent lamps in the near future. Recently, considerable efforts have been devoted to study phosphors used for white LEDs [3–5]. The rare earth ions activated inorganic phosphors have received much attention because of their wide applications in white LEDs, fluorescent lamps, display devices, solid-state lasers, biological labeling and soon [6,7]The Dy^{3+}, mainly as doping ions in phosphors, three two dominant bands in the emission spectrum. The yellow band (580nm) corresponds to the $^4F_{9/2} \rightarrow {}^6H_{13/2}$ transition while the blue band (481nm) corresponds to the $^4F_{9/2} \rightarrow {}^6H_{15/2}$ transition and red band (665nm) corresponds to the $^4F_{9/2} \rightarrow {}^6H_{11/2}$ transition [8, 9].The Dy doped phosphors have been also studied in terms of its long after glow white-emitting behaviors, but the reports on white LED phosphors doped with Dy^{3+} were quite rare. While the characteristic emission peaks of Dy^{3+} ions locate at blue light and yellow light regions.SrS, a member of AES family, acts as a good insulator due to indirect band gap of 4.2 eV in the bulk form. However, SrS doped with suitable activators such as rare earth ions exhibits excellent luminescent properties [10, 11]. Phosphor sample was prepared by the SSDM. SSDM is an effective and low-cost method for producing various industrially useful nanocrystalline materials. The diffusion process to prepare powder samples is very facile, which has been extensively applied to the preparation of various nano-sized sulfide materials [12]. This paper reports the structural characterization on the basis of XRD, FEGSEM, EDX and HRTEM analysis and studies of optical properties are also done on the basis of photoluminescence (PL).Till date there is no report published on Dy^{3+} doped SrS host; here we have reported for the first time by using solid state diffusion method .The prepared phosphors exhibits blue, yellow and red emission bands under near UV excitation wavelength, which is very much valuable for the production of white light.

EXPERIMENTAL

Sample Preparation

To synthesize the nanocrystallineDy^{3+}(0.5 mol%) doped SrS phosphors the solid state diffusion method was preferred [13]. The used ingredients were strontium sulfate, sodium thiosulfate, dysprosium oxide and activated charcoal, all of analytical grade (99.9 % pure). Strontium sulfate and activated charcoal were taken in a stoichiometric ratio, while sodium thiosulfate was 15% of the amount of strontium sulfate. All these materials were taken into an alumina crucible with a thin layer of carbon powder. The alumina crucibles were placed in a digitally controlled furnace for 2 hours already maintained at temperature of 1000°C and reaction started giving red hot flame. The solution of cerium nitrate in ethanol was preferred to maintain the uniform dispersion of activator within the starting material. In this method the reduction of sulfate using carbon powder into its sulfide form and incorporation of an activator in the lattice in the presence of a suitable flux at a high temperature takes place. As, it was over, the crucibles were pulled out of the furnace and kept in open to allow cooling. After cooling, the material was further ground using agate pestle and mortar to get powder form of phosphors.

Measuring Instruments

The materials were weighed using Shimadzu ATX 224 single pan analytical balance and the samples were prepared in a digital furnace. The crystalline structure, size and phase composition of the samples were examined by Bruker D8 Advance X-ray diffractometer using Cu-K_α radiation (λ = 1.54 A°). The morphology of prepared samples was studied using German make ZEISS-SUPRA 40 FESEM while energy dispersive X-ray spectroscopy was carried out with Oxford Inca EDX System. The morphology and size determination of the product were carried out by HRTEM using Model JEM 2100 equipped with a plate/CCD camera. Diluted nanoparticles suspended in acetone were introduced on a carbon coated copper grid and were allowed to dry in air for conducting HRTEM studies. All characterization studies were performed at room temperature (RT). Photoluminescence emission and

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excitation spectra were recorded by 5301 Spectrofluoro meter Shimadzu makes.

RESULTS AND DISCUSSION

XRD, FESEM, EDX & HRTEM studies

Figure 1 shows the X-ray powder diffraction (XRD) pattern of SrS: Dy^{3+} (pure and 0.5 mol% of Dy) phosphor.From the XRD pattern, the peak indexed revealed the pure cubic phase of SrS. When compared with International System for Diffraction Data (PDF card no.00-002-0659), these patterns are found to be characterized by peaks at 2θ values of 25º, 30º,42º,53º and 62º corresponding to the planes (111), (200), (220), (222) and (400). It is indicated that there is no impurity phase among the phosphor sample. From these experimental results we can conclude that Dy^{3+} ions have been introduced in to the SrS lattice, and do not cause any change in the cubic structure. It was revealed that the introduction of Dy^{3+} ions did not influence the crystal structure of the phosphor matrix. The crystallite size calculated by Scherer formula it confirms the formation of nano crystallites in phosphors SrS: Dy^{3+} (pure and 0.5 mol %).

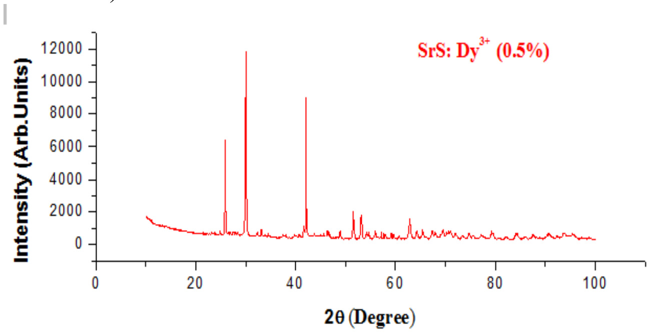


Figure 1: XRD of pure SrS: Dy^{3+} (0.5 mol %).

The FESEM has been used as a powerful tool to intensely demonstrate the sizes, surface morphologies and molecular structures of the samples in different forms [13]. The FESEM images of the SrS: Dy^{3+} (pure and 0.5%) phosphor samples are shown in Fig.2. Generally, the size and morphology of the particles depend on the various synthesis methods. In the present investigation, we preferred SSDM to synthesize the phosphors .The FESEM image of the SrS: Dy^{3+} phosphor sample observed that the structure of the phosphor is flake type nanostructure being semi-spherical in shape (figure 2). Sample shows formation of nano-structured and connectivity with grains over the surface. Here some agglomerates formation occurs due to high temperature synthesis method. Some particles are big and some are small as well as the particle distribution from nano to few microns size.

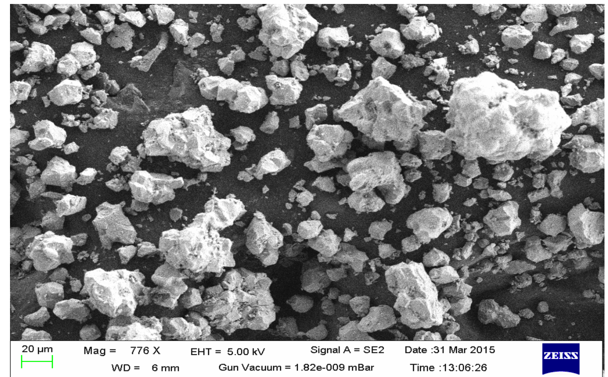


Fig. 2. FESEM image of SrS: Dy^{3+} (0.5mol %) phosphor.

The EDX qualitative and quantitative elemental analysis of SrS: Dy^{3+}phosphor sample is depicted in fig. 3. The result of EDX studies verifies the presence of strontium (Sr), sulphur (S) and dysprosium (Dy) in the prepared sample. Further, it is observed that the distribution of the elements is fairly uniform. Qualitative analysis of prepared sample was shown in table 1.

Table 1: EDX qualitative analysis of the SrS: Dy phosphor

S.no.	Elements	Atomic %
1.	Sr	45.39
2.	S	52.81
3.	Dy	1.80

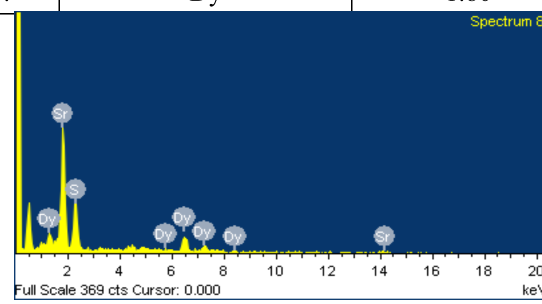
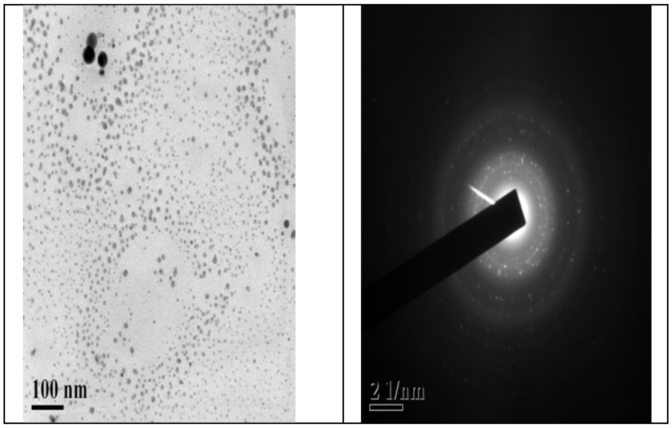


Fig.3: EDX spectra of SrS: Dy^{3+} (0.5mol %) phosphor

High resolution transmission electron microscopy (HRTEM) is used to study the shape, defects, grain and interface boundaries and calculate the crystallite size, d-spacing, etc [12]. The images revealed that the Dy^{3+} doped SrS powder was a collection of clustered100 nm distribution of particles (figure 4a). Synthesized sample showed the semi-irregular shapes, with no critical difference in morphology; this pattern is quite similar recorded by FEGSEM image. Semi-sphere formation is responsible for the very good PL spectra from prepared phosphor. Selected area electron diffraction (SAED) pattern of the phosphor shows some diffraction peaks which are accordance with XRD analysis of the sample (figure 4b).The diffraction dots in form of concentric rings seen in SAED pattern and signify the cubic phase of SrS displaying its single-crystal nature.



(a) (b)

Fig. 4: (a) HRTEM image of SrS: Dy^{3+} (0.5mol %) phosphor (b) SAED image of SrS: Dy^{3+} (0.5mol %) phosphor.

Photoluminescence spectra of SrS : Dy^{3+}

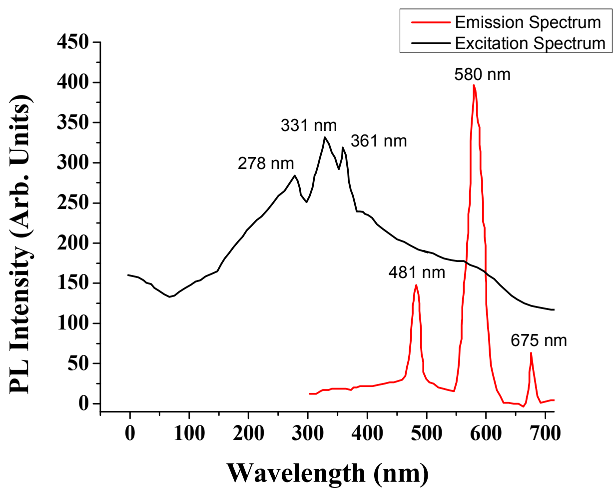


Fig. 5: PL of SrS: Dy^{3+} (0.5mol %) phosphor

In order to study the luminescent properties of phosphor, the excitation and emission spectrum of prepared SrS: Dy^{3+} phosphor was recorded. The emission spectrum of SrS: Dy shown in figure 5has three peaks which are observed at 481 nm,580 nm and 675 nm due to as $^4F_{9/2} \rightarrow {}^6H_{15/2}$, $^4F_{9/2} \rightarrow {}^6H_{13/2}$ and $^4F_{9/2} \rightarrow {}^6H_{11/2}$ transitions of Dy^{3+} ion respectively (excitation wavelength is 376 nm). The excitation spectrum was observed at 331 nm in between 200 and 400 nm, When the SrS: Dy^{3+} phosphor was excited at in the UV region, simultaneous emissions of blue, yellow and red nm position were observed. Notice that the red emission is less intense than the blue and yellow emissions. These three different emission bands originated from the one origin owing to their having the same excitation wavelength. The transitions involved in blue, yellow and red bands of Dy^{3+} ion are well known and have been identified as $^4F_{9/2} \rightarrow {}^6H_{15/2}$, $^4F_{9/2} \rightarrow {}^6H_{13/2}$ and $^4F_{9/2} \rightarrow {}^6H_{11/2}$ transitions respectively [14].

CONCLUSION

It is concluded that from the presented manuscript for dysprosium doped SrS phosphor is useful for the generation of white light emission give the PL emission peaks in intense blue yellow and red region. The phosphor can act single host with single doping for the formation of display materials and broad emission peaks. The characterization shows the cubic structure and surface morphology was good uniform and minimum agglomerates formation. The minimum agglomerate shows the good luminescence emissions.

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