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Effect of Flux on Photoluminescence Properties of Strontium Cerium Oxide Phosphor

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Abstract This paper report, Photoluminescence properties of the synthesized Sr_2CeO_4 phosphor using various fluxes by standard solid-state reaction method have been studied. Analytical grade inorganic salts like $Sr(NO_3)_2$, CeO_2 were used as raw materials of assay 99.9%. The required chemicals were weighed in molar ratio 2:1 and roughly 10mg of urea, citric acid, and glycine were added as fluxes, thoroughly mixed, and grounded in an agate mortar and pestle. The final mixtures were heated at 1200 ºC for 3 hours in a muffle furnace with a heating rate of 5°C/min. The samples were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), photoluminescence (PL) technique. The XRD study reveals the formation of the material mostly is in a single phase. The average crystallite size is calculated using Scherer's formula ($D=K.\lambda\backslash B\cos\theta$). PL emission spectrums of Sr_2CeO_4 phosphor with different fluxes were shows broad emission from 350 – 650nm was peaking at 470nm. This broadband is due to the $f \rightarrow t_1g$ transition of Ce^{4+}. SEM image looks like grains morphology with different sizes and shapes. The Commission International de l'Eclairage coordinates was calculated.

Keywords Photoluminescence; Solid-state reaction method; Phosphor; flux; FEDs

1. Introduction

Recently various phosphor materials have been actively investigated to improve their luminescent properties and to meet the development of different display and luminescence devices. Inorganic compounds doped with rare-earth ions form an important class of phosphors as they possess a few interesting characteristics such as excellent chemical stability, high luminescence efficiency, and flexible emission colors with different activators. In recent years, much attention has been focused on oxide-based luminescent materials due to their commercial applications in color television, fluorescent tube, X-ray phosphors, and FEDs [1, 2]. The search for blue phosphors is of particular importance because of the limited number of stable blue luminescent materials available. Danielson et al. [3] have reported a novel blue luminescent material Sr_2CeO_4 prepared by combinatorial material synthesis technique, which exhibits the emission peak at 485nm. Subsequently, several studies of this luminescent material were conducted, and some synthetic routes have been developed to prepare the Sr_2CeO_4 powders and films, including the traditional solid-state reaction method. [5-8] Furthermore, luminescent properties were found to greatly depend on particle size, size distribution, and particle morphology. This relationship is of current interest and may lead to new fabrication processes to yield high-quality luminescent materials. Therefore, the solid-state reaction

method is a useful and attractive technique for the preparation of oxide-based materials especially complex metal oxides [9-12].

2. Experimental Details

The phosphor samples are prepared via a solid-state reaction method (SSR). We prepared Sr_2CeO_4 phosphors by weighing, mixing inorganic salts, assay 99.9%, Strontium nitrate [Sr $(NO_3)_2$], Cerium oxide (CeO_2) in 2:1 molar ratio by adding 10mg of citric acid, urea (NH_2CONH_2), and Glycine (NH_2CH_2COOH) in each sample. We ground into a fine powder using an agate mortar and pestle about an hour. The samples were fired at 1200 ºC for 2 hours with a heating rate of 5 ^oC/min in a muffle furnace.

3. Results and Discussion

The crystalline structure of the powders was analyzed by X-ray powder diffraction (XRD). The XRD was done on Indus beam line-II at RRCAT, Indore, India. Fig.1 is the XRD pattern of Sr_2CeO_4 phosphors with urea and citric acid as fluxes. The same pattern was shown by the glycine and without flux samples, which were not shown in the figure. The crystalline phase was identified with the ICDD database card 89-5546 [4]. The majority of the diffraction peaks was well indexed and confirms the Sr_2CeO_4 single phase. It indicates that the heat treatment temperature and time were sufficient to form a single phase. The calculated crystallite size using Scherer's formula D=K.λ\Bcosθ, where k the constant (0.94), λ the wavelength of the X-ray (0.895Å), β the full-width at half maxima of Sr_2CeO_4 without flux, with glycine, with urea, and with citric acid are around ~9, 10, 11and 18nm respectively, and particle sizes are around ~26µm, 16µm, 22µm, 28 µm respectively.

Fig.2 shows the excitation and emission spectra of Sr_2CeO_4 powders recorded at room temperature. The excitation spectrum shows broadband with peaks around 250,270 and 280 nm respectively and the corresponding emission spectrum consists of broadband with a maximum at 470nm. These bands in the excitation and emission spectra have been assigned to the $Ce^{4+} – O^{2-}$ charge-transfer transition [15]. Considering that an electron can be transferred from an oxygen ligand to the empty 4f shell of Ce^{4+}, a high spin-triplet excited state is formed via a spin-forbidden transition. Therefore, the PL can be assigned to a ligand-to-metal charge-transfer transition of Ce^{4+} [16]. In Sr_2CeO_4, two kinds of Ce^{4+} ions exist, i.e two different $Ce^{4+}– O^{2-}$ bond lengths in the lattice [15]. The two excitation bands observed in the figure are thus attributed to two different charge-transfer transitions.

From fig.2, it is observed that Sr_2CeO_4 with various fluxes shows a broad emission band from 350–650nm with a peak at 470nm under 270nm excitation. This broadband is due to $f \rightarrow t_1g$ transitions of Ce^{4+}. However urea is used as a flux, the intensity of 470nm emission increased by 500% in comparison with pure Sr_2CeO_4. Surprisingly when citric acid is used as a flux, the intensity of 470nm emission is increased by 600% in comparison with pure Sr_2CeO_4. It is concluded that citric acid plays a major role in the formation of Sr_2CeO_4 phosphor.

Fig.3 shows the SEM image of Pure Sr_2CeO_4, glycine, Urea, and Citric acid as flux. The sample exhibits grain like morphology with different sizes and shape. At low magnification, the particles appear agglomerated and at high enough magnification, the nature of individual crystallites is evident. Particle size is estimated through this analysis as uniformity of the particle shape and size affects the luminescence efficiency of phosphor materials. From the SEM images of Sr_2CeO_4 with different fluxes reveal that particles are agglomerated and have size estimation all are in 5µm range which would be helpful for display device technological applications.

The CIE coordinates of (chart -1931) were calculated by the Spectrophotometric method using the spectral energy distribution of the Pure Sr_2CeO_4 and different fluxes were added to Sr_2CeO_4 shown in Fig.4. The color coordinates for the un-doped Sr_2CeO_4 are (a) x = 0.208 and y = 0.28. The color coordinates for the Sr_2CeO_4 with glycine as flux are (b) x = 0.208 and y = 0.29. The color coordinates for the Sr_2CeO_4 with urea as flux are (c) x = 0.208 and y = 0.269. The color coordinates for the Sr_2CeO_4 with citric acid as flux are (d) x = 0.208 and y = 0.258. This is another positive feature of the sample synthesized using the solid-state reaction method, getting good color coordinates[13-17], this in turn leads to better chromaticity, hence improving the overall color rendering index of the phosphors.

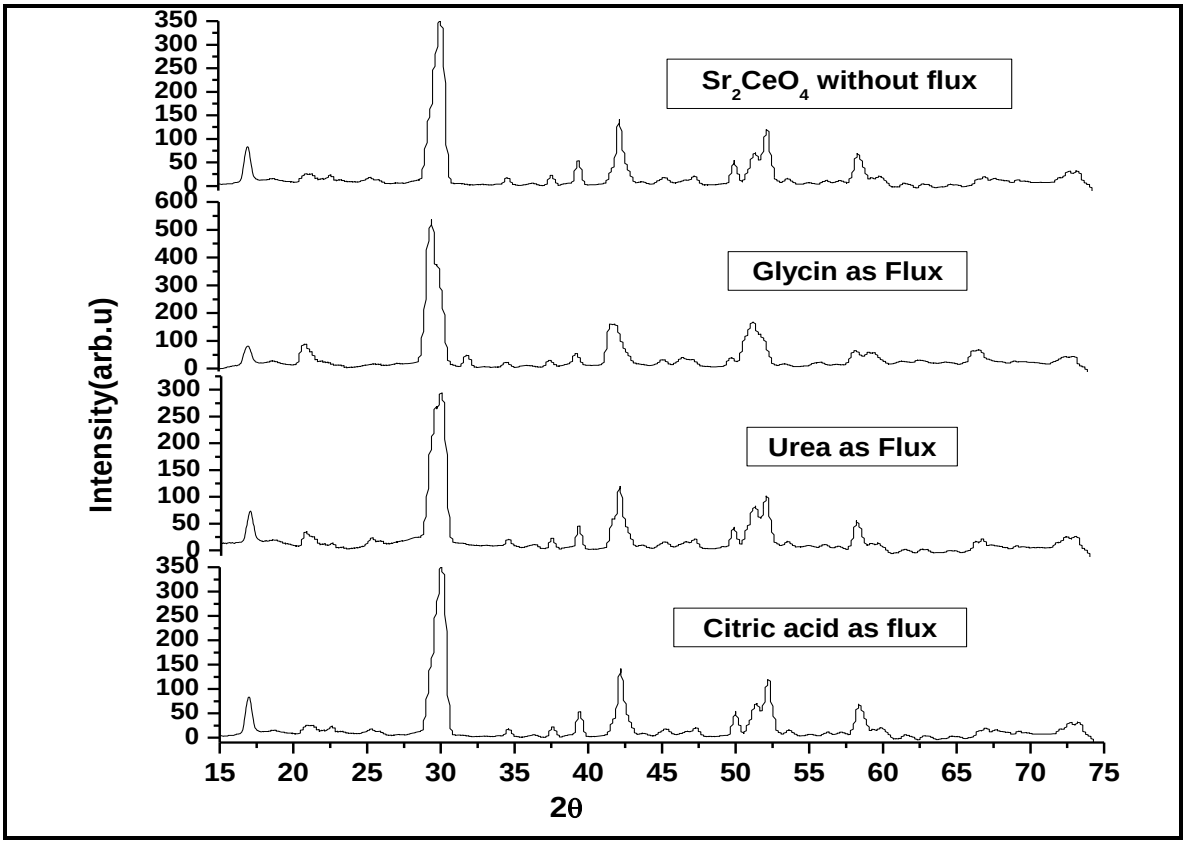

Figure 1: X-ray powder diffraction pattern of Sr_2CeO_4 with different Fluxes

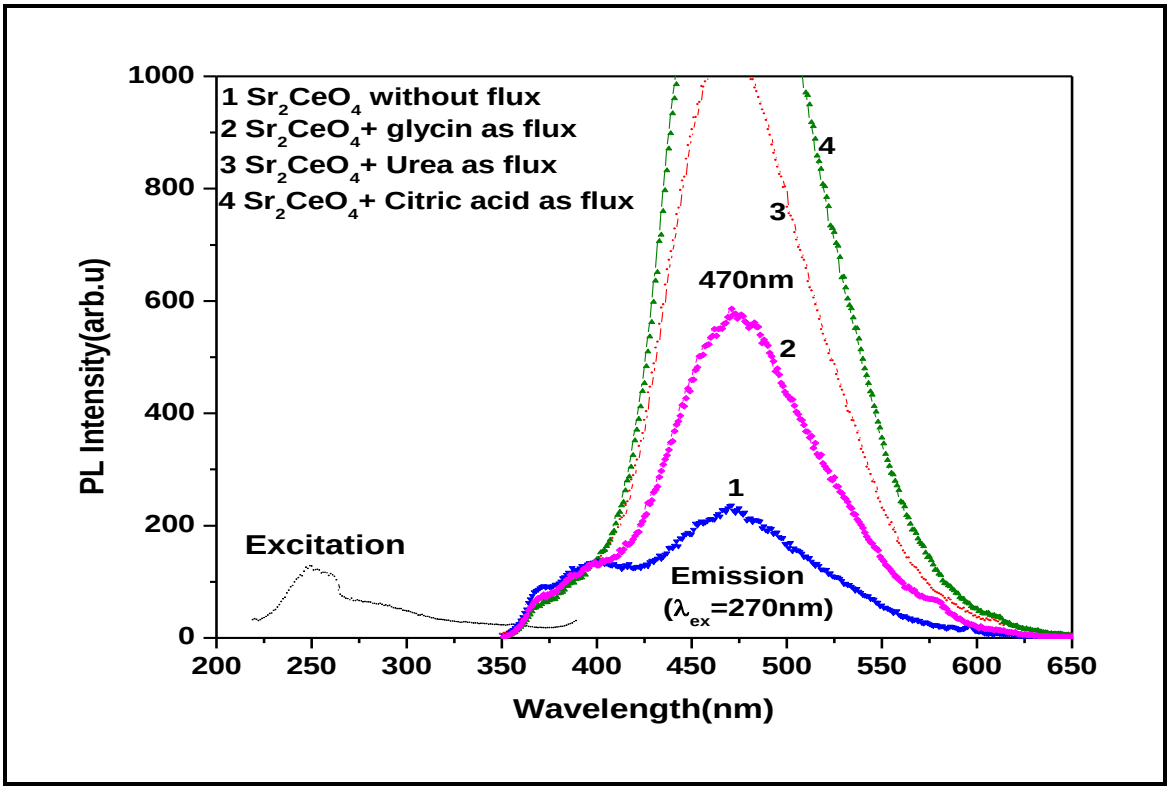

Figure 2: Excitation and emission spectrum of Sr_2CeO_4 with different fluxes

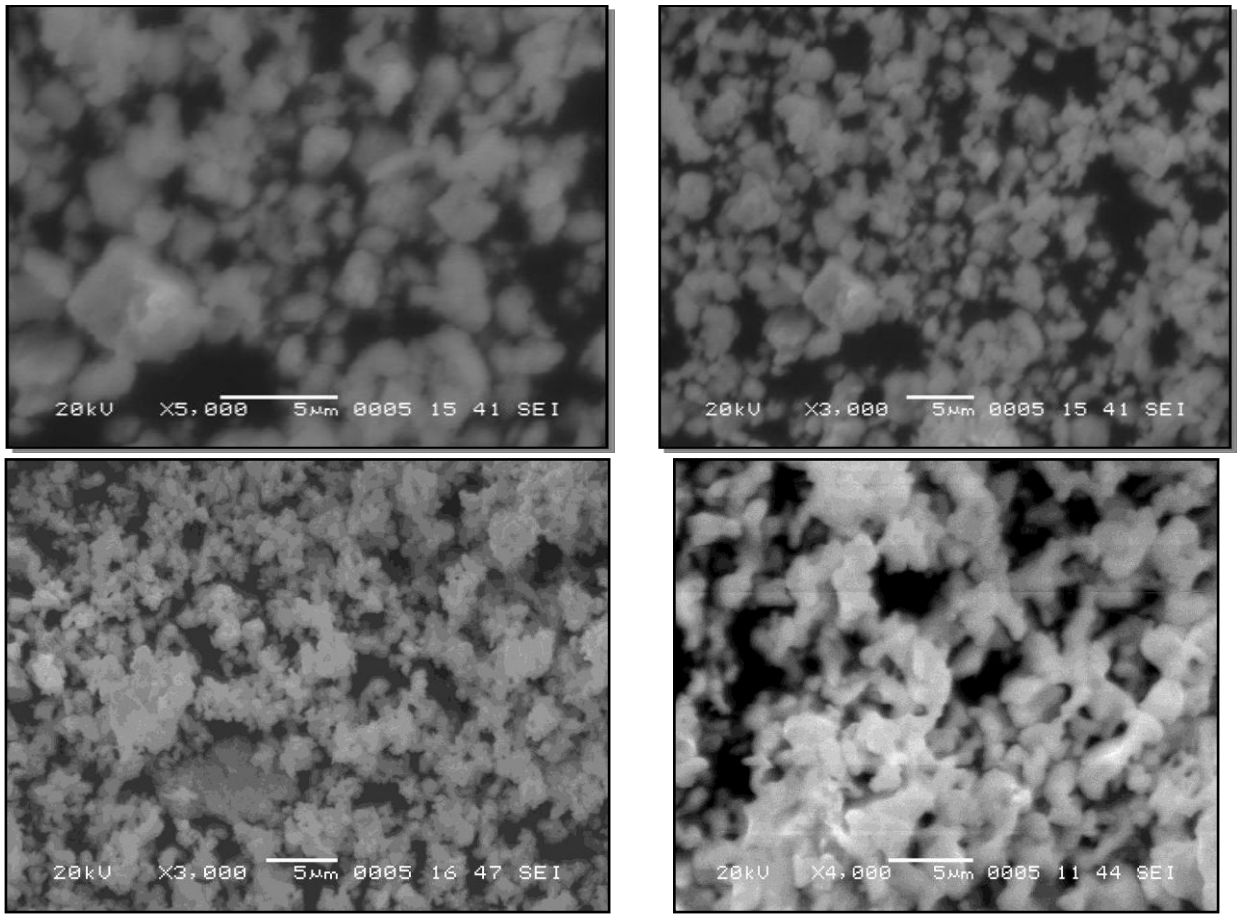

Figure 3: SEM images of Sr_2CeO_4 with different fluxes

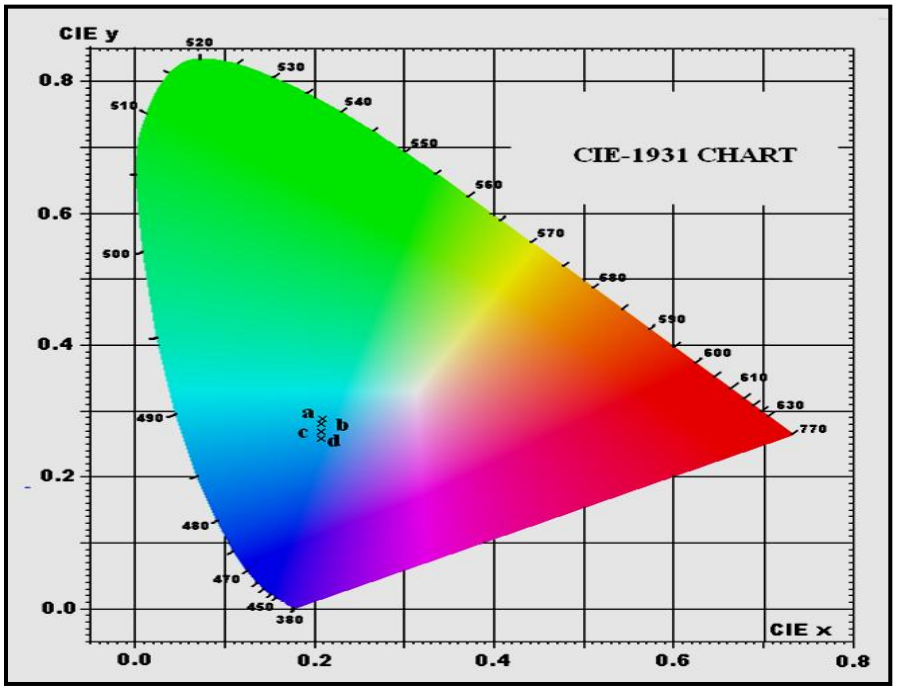

Figure 4: CIE Coordinates of Sr_2CeO_4 with different fluxes

4. Conclusions

- From fig.2, it is observed that Sr_2CeO_4 with various fluxes shows a broad emission band from 350–650nm with a peak at 470nm under 270nm excitation. This broadband is due to $f \rightarrow t_1g$ transitions of Ce^{4+}. However urea is used as a flux, the intensity of 470nm emission increased by 500% in comparison with pure Sr_2CeO_4. Surprisingly when citric acid is used as a flux, the intensity of 470nm emission is increased by 600% in comparison with pure Sr_2CeO_4. It is concluded that citric acid plays a major role in the formation of Sr_2CeO_4 phosphor.
- From the SEM images of Sr_2CeO_4 with different fluxes reveal that particles are agglomerated and have size estimation all are in 5µm range which would be helpful for display device technological applications.
- From the CIE colour coordinates of (chart -1931) this phosphor is another positive feature of the sample synthesized using the solid-state reaction method, getting good color coordinates, this in turn leads to better chromaticity, hence improving the overall color rendering index of the phosphors.

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