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Photoluminescence study of rare earth ions doped Sr_2CeO_4 phosphor prepared via conventional solid- state reaction method

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Abstract

The lanthanide ions either in their divalent or trivalent charge state form a very important class of luminescence activators in phosphors and single crystals. Doping these lanthanide ions in different host lattices are widely utilized in many areas for example the 5d-4f emission of Ce^{3+} used in scintillators for γ -ray detection, cathode ray tubes and electroluminescence phosphors. The photon cascade emission from 4f² levels of Pr^{3+} used for developing high quantum efficiency phosphors excited by means of a Xe discharge in the vacuum-UV. The narrow line 4f³ transition in Nd^{3+} is used in laser crystals. The Sm^{3+} emission used as an electron trap and in information storage properties like optical memory applications, X-ray imaging phosphor applications. The famous $^5\text{D}_0$ - $^7\text{F}_j$ 4f⁶ redline emission of Eu^{3+} and the blue to red 5d-4f emission of Eu^{2+} are used in display and lighting phosphors. The 4f⁸ line emission of Tb^{3+} is used in tricolor tube lighting. The Dy^{3+} emission plays an important role in the persistent luminescence phosphors. Er^{3+} , Tm^{3+} used to investigate for possible photon cascade emission phosphor applications. This is brief and still incomplete summary illustrates the diversity of applications involving the luminescence of lanthanide ions.

Keywords: Strontium cerium oxide (Sr_2CeO_4) phosphor, rare earth ion, conventional solid-state reaction method

1. Introduction

The trivalent ions of the lanthanide series are characterized by a gradual filling of the 4f orbital, from 4f⁰ (for La^{3+}) to 4f¹⁴ (for Lu^{3+}) and shown in table 1. One of the most interesting features of these ions is their photoluminescence. Several lanthanide ions show luminescence in the visible or near-infrared spectral regions upon irradiation with ultraviolet radiation. The color of the emitted light depends on the lanthanide ion. For instance, Eu^{3+} emits red light, Tb^{3+} green light, Sm^{3+} orange light, and Tm^{3+} blue light. Yb^{3+} , Nd^{3+} , and Er^{3+} are well-known for their near-infrared luminescence, but other lanthanide ions (Pr^{3+} , Sm^{3+} , Dy^{3+} , Ho^{3+} , and Tm^{3+}) also show transitions in the near-infrared region. Gd^{3+} emits in the ultraviolet region, but its luminescence can only be observed in the absence of organic ligands with low-lying singlet and triplet levels [1-3]. When the light emission by lanthanide ions is discussed, one often uses the term 'luminescence', rather than the terms 'fluorescence' or 'phosphorescence'. The emission mechanism: *fluorescence* is singlet-to-singlet emission (i.e., a spin-allowed transition) and *phosphorescence* is triplet-to-singlet emission (i.e., a spin forbidden transition). The lanthanide rare earths' electronic configuration consists of a filled xenon core and 6s shells, followed by filling of 5d and 4f shells. The interesting feature of the lanthanide series is that their 4f shell begins to fill after 6s. In addition, the 4f shell, instead of having a radius larger than the 5s and 5p shells, actually contracts and becomes bounded by these shells. The trivalent state is formed by removal of two of the 6s electrons and one of the 4f electrons, with the outer 5s and 5p shells remaining intact [4-7]. This configuration renders the 4f electronic levels less sensitive to the glass or crystal host site environment. The optical spectrum of the trivalent ions of lanthanide rare earths consists of various excited states of the 4f electrons. The spread in the 4f^N transitions spectra arises from various atomic interactions between the electrons, and Stark splitting due to the host environment [8-12]. In the case of the lanthanides, the emission is due to transitions inside the 4f shell, thus intra-configurational f-f transitions. Because the partially filled 4f shell is well shielded from its environment by the

closed $5s^2$ and $5p^6$ shells, the ligands in the first and second co-ordination sphere perturb the electronic configurations of the trivalent lanthanide ions only to a very limited extent. This shielding is responsible for the specific properties of lanthanide luminescence, more particularly for the

narrowband emission and for the long lifetimes of the excited states. Ce^{3+} is a special case because this ion emits intense broadband emission due to allowed f-d transitions. The position of the emission maximum strongly depends on the ligands environment of the Ce^{3+} ion^[9-15, 26].

Table 1: Electronic structure of the trivalent lanthanide ions

| Element | Symbol | Atomic number (Z) | Configuration Ln^{3+} | Ground state Ln^{3+} |
|--------------|--------|-------------------|-------------------------|------------------------|
| Lanthanum | La | 57 | $[Xe]4f^0$ | 1S_0 |
| Cerium | Ce | 58 | $[Xe]4f^2$ | $^2F_{5/2}$ |
| Praseodymium | Pr | 59 | $[Xe]4f^3$ | 3H_4 |
| Neodymium | Nd | 60 | $[Xe]4f^4$ | $^4I_{9/2}$ |
| Promethium | Pm | 61 | $[Xe]4f^5$ | 5I_4 |
| Samarium | Sm | 62 | $[Xe]4f^6$ | $^6H_{5/2}$ |
| Europium | Eu | 63 | $[Xe]4f^7$ | 7F_0 |
| Gadolinium | Gd | 64 | $[Xe]4f^8$ | $^8S_{7/2}$ |
| Terbium | Tb | 65 | $[Xe]4f^9$ | 7F_6 |
| Dysprosium | Dy | 66 | $[Xe]4f^{10}$ | $^6H_{15/2}$ |
| Holmium | Ho | 67 | $[Xe]4f^{11}$ | 5I_8 |
| Erbium | Er | 68 | $[Xe]4f^{12}$ | $^4I_{15/2}$ |
| Thulium | Tm | 69 | $[Xe]4f^{13}$ | 3H_6 |
| Ytterbium | Yb | 70 | $[Xe]4f^{14}$ | $^2F_{7/2}$ |
| Lutetium | Lu | 71 | $[Xe]4f^{14}$ | 1S_0 |

2. Experimental work

The present paper reports on the synthesis and characterization of rare earth ions doped Sr_2CeO_4 phosphor, using solid state reaction method. The compounds like Strontium Carbonate ($SrCO_3$), Cerium Oxide (CeO_2) and (La^{3+} , Pr^{3+} , Nd^{3+} , Sm^{3+} , Eu^{3+} , Gd^{3+} , Tb^{3+} , Dy^{3+} and Er^{3+}) (Sigma-Aldrich Chemie Inc., Germany) in the oxide form of high purity of analytical grade exceeding (99.9% assay) chemicals were used as starting materials. First we prepared Sr_2CeO_4 phosphor, without adding any dopants, as host material by weighing, adding Strontium carbonate ($SrCO_3$) (Sigma-Aldrich Chemie Inc., Germany), Cerium Oxide (CeO_2) (National Chemicals, Vadodara, India) were used as starting materials for the host material taken in stoichiometric proportions of Sr: Ce as 2:1 and ground into a fine powder using agate mortar and pestle about an hour. The grounded sample was placed in an alumina crucible and fired at $1200^\circ C$ for 3 hours in a muffle furnace with a heating rate of $5^\circ C/min$. The sample is allowed cool to room temperature in the same furnace for about 20 hours. In the same way rare earth (RE) ions (La^{3+} , Pr^{3+} , Nd^{3+} , Sm^{3+} , Eu^{3+} , Gd^{3+} , Tb^{3+} , Dy^{3+} and Er^{3+}) are doped at 0.5mol% concentration only.

We investigated the phase purity, morphology, Photoluminescence excitation and emission and CIE color coordinates. XRD analysis was carried out with a powder diffractometer (Rigaku-D/max 2500) using Cu $K\alpha$ radiation, microstructures/morphology of the samples were studied using a scanning electron microscopy (Philips XL-CP-30), Photoluminescence emission and excitation spectra were measured by Spectro fluorophotometer (SHIMADZU, RF-5301 PC) using Xenon lamp as excitation source, All the spectra were recorded at room temperature. Emission and excitation spectra were recorded using a spectral slit width of 1.5nm. The Commission International de l'Eclairage (CIE-1931 chromaticity diagram) color co-ordinates were calculated by the spectrophotometric method using the spectral energy distribution. The chromatic color coordinates

(x, y) of prepared materials was calculated with colour calculator version 2, software from Radiant Imaging^[17].

3. Results and Discussion

3.1 XRD Analysis

X-ray powder diffraction (XRD) of un-doped Sr_2CeO_4 phosphor was carried out at room temperature. Fig.1 shows XRD Pattern of un-doped Sr_2CeO_4 phosphor. From the XRD pattern analysis it was found that the prominent phase formed is Sr_2CeO_4 phosphor, after the computer program POWD (an interactive Powder diffraction Data Interpretation and Indexing Program, Version 2.2) was used to calculate hkl and d values were found to be in good agreement with the JCPDS data card No. 50-0115. This reveals that the structure of Sr_2CeO_4 is orthorhombic and is in agreement with the findings of the previous workers like Sankar *et al.*^[18], Danielson *et al.* However the data reported by Jiang *et al.*^[20] and Serra *et al.*^[21] indicate triclinic structure. It clearly indicates that the heat treatment temperature and time of cooling were sufficient to form single phase. The calculated lattice parameters are $a = 6.094 \text{ \AA}$, $b = 10.232 \text{ \AA}$, $c = 3.566 \text{ \AA}$, and volume $V = a*b*c = 6.094*10.232*3.566 = 222.354 \text{ \AA}^3$. The crystallite size was determined using the Scherrer's equation $D = k\lambda / \beta \cos \theta$, where k the constant (0.94), λ the wavelength of the X-ray (0.154 nm or 1.54 \AA), β the full-width at half maxima (FWHM) and θ the Bragg angle of the XRD peak (1,1,1). The calculated average crystallite size of un-doped Sr_2CeO_4 is $\sim 28 \text{ nm}$.

Fig.2 shows XRD pattern of rare earth (RE^{3+}) ions (La^{3+} , Pr^{3+} , Nd^{3+} , Sm^{3+} , Eu^{3+} , Gd^{3+} , Tb^{3+} , Dy^{3+} and Er^{3+}) doped Sr_2CeO_4 phosphor at 0.5mol% concentration. All the diffraction peaks are looking same as un-doped Sr_2CeO_4 phosphor, concluding that the dopant ions did not affect the host structure except the increase or decrease of intensity. The calculated average crystallite sizes of Sr_2CeO_4 doped RE^{3+} phosphors are shown in the table. 2.

Table 2: The calculated average crystallite sizes of Sr₂CeO₄ doped RE³⁺ phosphors

| S. No. | Rare earth ion | Average Crystallite size (nm) |
|--------|---|-------------------------------|
| 1 | Un-doped Sr ₂ CeO ₄ | 28 |
| 2 | Sr ₂ CeO ₄ : La ³⁺ | 23 |
| 3 | Sr ₂ CeO ₄ : Pr ³⁺ | 25 |
| 4 | Sr ₂ CeO ₄ : Nd ³⁺ | 29 |
| 5 | Sr ₂ CeO ₄ : Sm ³⁺ | 30 |
| 6 | Sr ₂ CeO ₄ : Eu ³⁺ | 31 |
| 7 | Sr ₂ CeO ₄ : Gd ³⁺ | 23 |
| 8 | Sr ₂ CeO ₄ : Tb ³⁺ | 26 |
| 9 | Sr ₂ CeO ₄ : Dy ³⁺ | 29 |
| 10 | Sr ₂ CeO ₄ : Er ³⁺ | 22 |

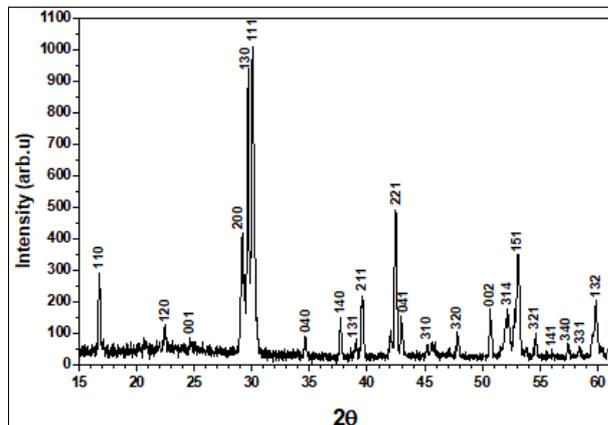


Fig 1: XRD Pattern of un-doped Sr₂CeO₄ phosphor

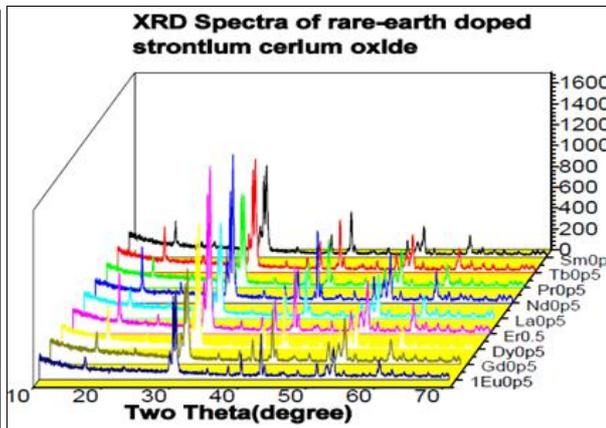


Fig 2: XRD Pattern of RE³⁺ ions doped Sr₂CeO₄ phosphor

3.2 Photoluminescence Study

The excitation and emission spectra recorded for un-doped Sr₂CeO₄ at room temperature are presented in Fig.3. The excitation spectrum monitored at ~400 nm shows a broad band with one maximum at 280 nm. The feature of the excitation spectrum changed when monitored at ~ 470 nm shows a broad band with two maxima at 280 and 350 nm. These bands have been assigned as intra configurationally Ce⁴⁺- O²⁻ charge transfer (CT) transitions by Danielson *et al.* [22]. The emission spectra recorded on exciting at 254, 260, 280 as well as 350 nm show a broad band with the maximum in the blue-green region (FWHM~86) with extended emission into longer wavelengths. It is well known that the CT transition is sensitive to ligand environment i.e. the potential field of the ligands. In order to find out how the next-nearest-neighbor (NNN) of the central metal ion Ce⁴⁺, affects the Ce⁴⁺- O²⁻ CT transition, Sr₂CeO₄ phosphor has been doped with other rare-earth (RE) ions e.g. La³⁺, Pr³⁺,

Nd³⁺, Sm³⁺, Eu³⁺, Gd³⁺, Tb³⁺, Dy³⁺ and Er³⁺. These ions have unique CT associated excited states in their complexes with halogen ligands or in oxides.

Sr₂Eu_xCeO₄ with low concentrations of Eu³⁺ (x=1.5mol %) showed white emission whereas red luminescence occurred at higher concentrations (x= >3). The excitation spectra recorded by fixing the highest intensity Eu³⁺ emission as the monitoring wavelength show the same features for all the compositions. Fig.4 presents the excitation and emission spectra for the composition (RE x=0.5mol %). The spectra show a broad band peaking at 280 nm and a broad and sharp shoulder at 350 nm, which are already assigned to the Ce⁴⁺- O²⁻ CT transition in un-doped Sr₂CeO₄ phosphor [23]. In addition, the sample shows the characteristic intra configuration 4f – 4f transitions of the Eu³⁺ ion. ⁷F₀ → ⁵L₆ transition at 395nm and ⁷F₀ → ⁵D₂ transition at 467nm, of these two lines 467nm is the strongest and well matched with the blue LED chip.

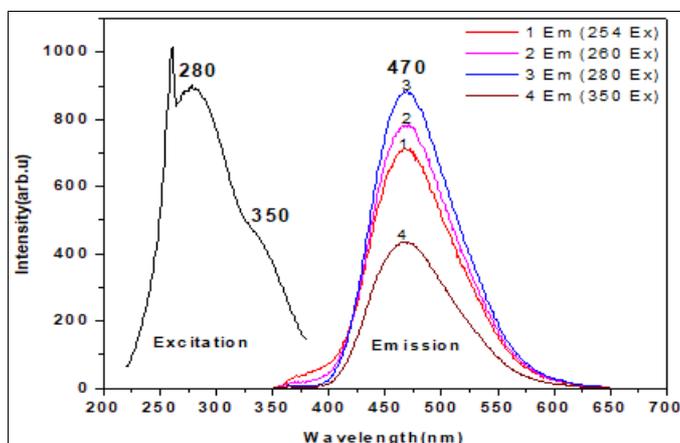


Fig 3: XRD Pattern of un-doped Sr₂CeO₄ phosphor

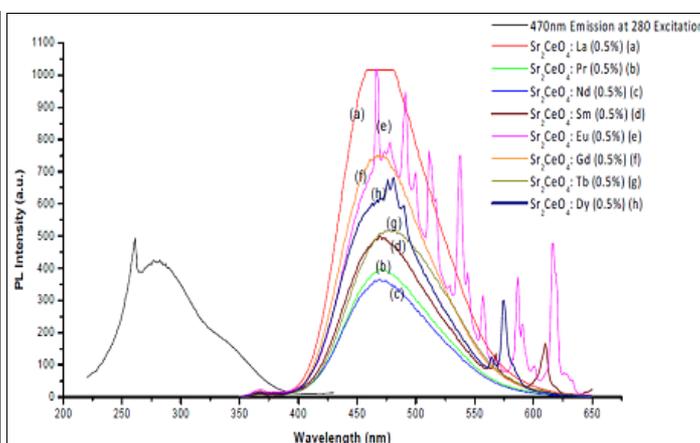


Fig 4: Excitation & Emission spectrum of RE³⁺ ions doped Sr₂CeO₄ phosphor

The emission spectrum of RE^{3+} ion doped Sr_2CeO_4 phosphor is composed of a broad band of lower intensity in the green-blue spectral region (which is already observed in the undoped Sr_2CeO_4 phosphor) that overlaps with a set of Eu^{3+} intra- $4f_6$ sharp lines in the blue, green and red regions. They are assigned to the transitions between the $^5\text{D}_0$, $^5\text{D}_1$ and $^5\text{D}_2$ excited states and $^7\text{F}_{0-3}$ ground level multiplets (Table. 2). The observed intra- $4f_6$ transitions are mainly of electric-dipole (ED) nature except the $^5\text{D}_0 \rightarrow ^7\text{F}_1$, $^5\text{D}_1 \rightarrow ^7\text{F}_0$ and $^5\text{D}_2 \rightarrow ^7\text{F}_1$ lines, which have predominant magnetic-dipole (MD) contribution. The low intensity of $^5\text{D}_0 \rightarrow ^7\text{F}_0$ line is because of the fact that this transition is forbidden by both ED as well as MD

selection rules. According to the MD transition is relatively independent of the kind of ligands as well as the symmetry of the coordination polyhedron around the lanthanide ions [24]. The intensities of the induced ED transitions in lanthanide ions are not much affected by the environment. However the transitions, which are very sensitive to the environment, are known as hypersensitive transitions. For Eu^{3+} , $^5\text{D}_2 \rightarrow ^7\text{F}_0$, $^5\text{D}_1 \rightarrow ^7\text{F}_1$ and $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transitions are indicated as hypersensitive by the relative intensities of the MD transitions remain the same for all the compositions. However, drastic changes are noticed in the relative intensities of the hypersensitive transitions.

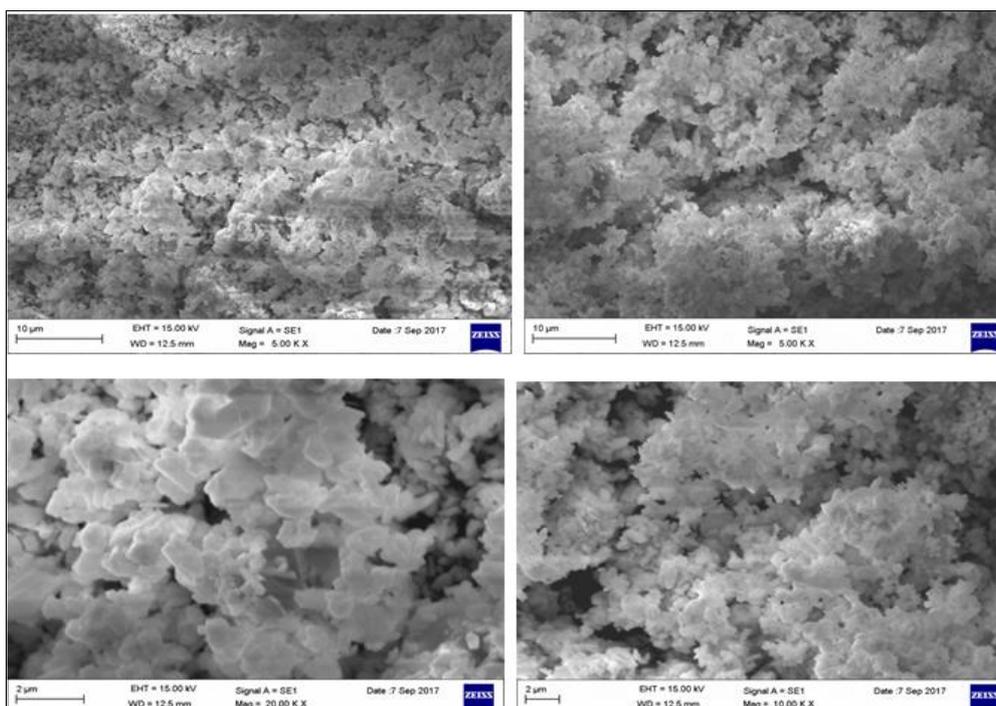
Table 3: Transitions of RE^{3+} ions doped in Sr_2CeO_4 Phosphor

| S. No | Dopant | λ (nm) | I_{emi} | Transitions |
|-------|--------|----------------|------------------|--|
| 1. | La | 467 | 1005 | $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$ |
| 2. | Pr | 467 | 755 | $^3\text{P}_0 \rightarrow ^3\text{H}_4$ |
| 3. | Nd | 467 | 635 | $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{9/2}$ |
| 4. | Gd | 469 | 535 | $^6\text{P}_{7/2} \rightarrow ^8\text{S}_{7/2}$ |
| 5. | Tb | 475 | 395 | $^5\text{D}_4 \rightarrow ^7\text{F}_6$ |
| 6. | Sm | 565 | 165 | $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{5/2}$ |
| | | 610 | 222 | $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ |
| 7. | Dy | 480 | 936 | $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2}$ |
| | | 564 | 191 | $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{13/2}$ |
| | | 573 | 526 | $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{11/2}$ |
| 8. | Eu | 467 | 1020 | $^5\text{D}_2 \rightarrow ^7\text{F}_0$ |
| | | 491 | 1020 | $^5\text{D}_2 \rightarrow ^7\text{F}_2$ |
| | | 513 | 892 | $^5\text{D}_2 \rightarrow ^7\text{F}_3$ |
| | | 538 | 923 | $^5\text{D}_1 \rightarrow ^7\text{F}_1$ |
| | | 556 | 372 | $^5\text{D}_1 \rightarrow ^7\text{F}_2$ |
| | | 587 | 465 | $^5\text{D}_0 \rightarrow ^7\text{F}_1$ |
| | | 617 | 540 | $^5\text{D}_0 \rightarrow ^7\text{F}_2$ |

3.3 Scanning Electron Microscopy study

Fig.5 presents the scanning electron microscopy (SEM) images of un-doped and RE^{3+} (0.5mol %) ions doped Sr_2CeO_4 Phosphor. The entire sample exhibits grain like morphology with different sizes and shape. At low and high enough magnification the particles appear agglomerated; 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 [25]. From the SEM images of un-doped and RE^{3+} (0.5mol %) ions doped Sr_2CeO_4 Phosphor reveal that particles are highly agglomerated and non-uniform size estimated particles are appeared. However all the Bessel diameters are around in 10-2 μm range; which can be helpful for display device technological applications in LEDs.



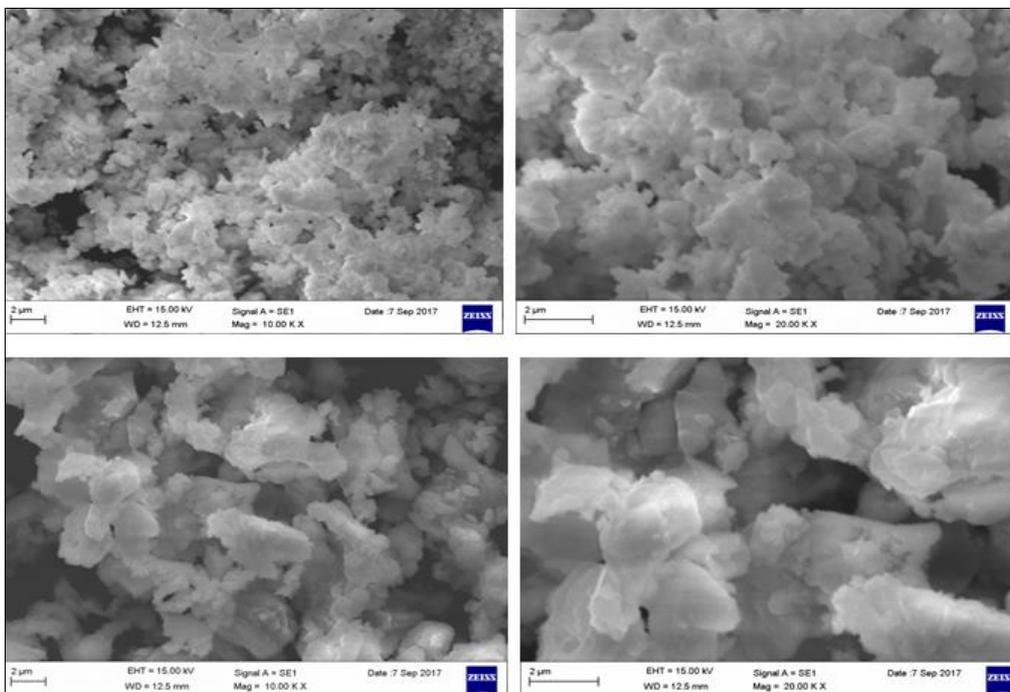


Fig 5: SEM images of un-doped & RE³⁺ (0.5mol %) ions doped Sr₂CeO₄ Phosphor

3.4 CIE Coordinates (1931 Chart)

Fig.6 Shows the CIE Colour coordinates of (1931chart) the un-doped and RE³⁺ (0.5mol %) ions doped Sr₂CeO₄ Phosphor. The present CIE Colour coordinates of (1931chart) the un-doped and RE³⁺ (0.5mol %) ions doped Sr₂CeO₄ Phosphor emitting with different colours like as blue, green, yellow and red regions under the excitation of 280, 350 nm respectively.

The CIE chromaticity coordinates for the un-doped and RE³⁺

(0.5mol %) ions doped Sr₂CeO₄ Phosphor excited at different wavelengths are summarized in table.3. The un-doped and RE³⁺ (0.5mol %) ions doped Sr₂CeO₄ Phosphors can be utilized for many applications. These single host lattices emitting white light under UV and nUV excitations is an interesting phenomenon. Hence, these phosphors are multifunctional phosphors, which one can use according to the need.

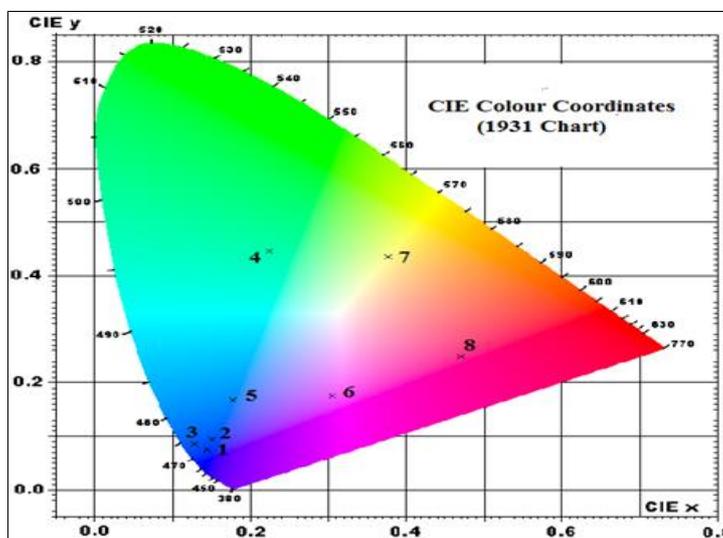


Fig 6: CIE Chromaticity diagram of un-doped & RE³⁺ (0.5mol %) ions doped Sr₂CeO₄ Phosphor

Table 4: Show the coordinates and colour region

| S. No | Dopant | Coordinates | | Colour Region |
|-------|--------|-------------|-------|---------------|
| | | X | Y | |
| 1. | La | 0.151 | 0.092 | Blue |
| 2. | Pr | 0.145 | 0.075 | Blue |
| 3. | Nd | 0.128 | 0.086 | Deep Blue |
| 4. | Gd | 0.224 | 0.445 | Green |
| 5. | Tb | 0.178 | 0.168 | Blue |
| 6. | Sm | 0.305 | 0.177 | Bluish Red |
| 7. | Dy | 0.377 | 0.435 | Yellow |
| 8. | Eu | 0.470 | 0.250 | Red |

4. Conclusions

- From the XRD pattern analysis it was found that the prominent phase formed is Sr₂CeO₄ phosphor, after the computer program POWD were found to be in good agreement with the JCPDS data card No. 50-0115.
- This reveals that the structure of Sr₂CeO₄ is orthorhombic and is in agreement with the findings of the previous workers.
- The excitation spectrum monitored at ~400 nm shows a broad band with one maximum at 280 nm.

- The feature of the excitation spectrum changed when monitored at ~ 470 nm shows a broad band with two maxima at 280 and 350 nm.
- These bands have been assigned as intra configurationally $Ce^{4+} - O^{2-}$ charge transfer (CT) transitions.
- The emission spectra recorded on exciting at 254, 260, 280 as well as 350 nm show a broad band with the maximum in the blue-green region (FWHM~86) with extended emission into longer wavelengths.
- The emission spectrum of RE^{3+} ion doped Sr_2CeO_4 phosphor is composed of a broad band of lower intensity in the green-blue spectral region (which is already observed in the un-doped Sr_2CeO_4 phosphor) that overlaps with a set of Eu^{3+} intra- $4f_6$ sharp lines in the blue, green and red regions.
- Fig.5 presents the scanning electron microscopy (SEM) images of un-doped and RE^{3+} (0.5mol %) ions doped Sr_2CeO_4 Phosphor. The entire sample exhibits grain like morphology with different sizes and shape.
- The present CIE Colour coordinates of (1931chart) the un-doped and RE^{3+} (0.5mol %) ions doped Sr_2CeO_4 Phosphor emitting with different colours like as blue, green, yellow and red regions under the excitation of 280, 350 nm respectively.
- These single host lattices emitting white light under UV and nUV excitations is an interesting phenomenon. Hence, these phosphors are multifunctional phosphors, which one can use according to the need.

5. Acknowledgment

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