

Synthesis, Characterizations and Luminescence Study of Erbium Doped Sr₂CeO₄ Nano Phosphor

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Abstract: Sr₂CeO₄: Er³⁺ Phosphor was synthesized by high temperature Solid state reaction method. Using oxides as a raw materials and Erbium was used as an activator. XRD, SEM, EDAX and Photoluminescence spectra were using to investigation the formation process, microstructure and luminescence properties of the synthesized phosphor. The result shows orthorhombic structure and average crystallite size is 35 nm. The excitations are recorded by monitoring 470nm wavelength. All the phosphors are excited with 254, 275 and 340nm. When the phosphors are excited with various excitations, the basic Sr₂CeO₄ emission is found and the descending side i.e. 500 – 600nm contains the overlapped spiky emissions at 470, 527, 536, 545 and 558nm are clearly seen which the Er emissions are basically. The excitation spectra of Sr₂CeO₄: Er³⁺ phosphor display broad band with two peaks the former peak is stronger than the later one; this broad band is due to the charge transfer band of the Ce⁴⁺ ion. The emission spectrum of Sr₂CeO₄: Er³⁺ phosphor shows broad band spectra with peak around 470nm with the excitation by a radiation of 262nm. The emission of Sr₂CeO₄: Er³⁺ for the excitation 262nm was emits bluish Green light and the emission spectra shows a broad band with peak around at 470, 527 536, 545, and 558 nm.

Keywords: Photoluminescence, Solid state reaction method, XRD, SEM, Phosphor.

1. INTRODUCTION

The research for oxide phosphors has been increasing due to their applications in many fields, such as cathode ray tubes (CRTs), light-emitting diodes (LEDs) and field emission displays (FEDs). Rare earth applications in the field of display devices still a hot topic much of the research around the globe is to improve the phosphor efficiency and to enhance the luminescence properties of the phosphor with discovery of blue light emitting Sr₂CeO₄ by combinatorial chemistry method in 1998 by Danielson [1]. Sr₂CeO₄ consist of infinite edge-shearing CeO₆ octahedral chains separated by Sr atoms [2]. The luminescence originates from a ligand-to metal Ce⁴⁺ charge transfer [3]. The broad emission band is suitable for the doping of rare earth ions in pursuing new luminescent materials. The blue phosphors are very few and if a suitable blue phosphor is found then it can be added to the well studied red and green combination for white light emission from the phosphor. If blue phosphor Sr₂CeO₄ doped with trivalent rare earths europium and samarium emit in the red region of the visible spectra [4-9]. The rare earth materials exhibits excellent sharp-emission luminescence properties with suitable sensitization and effectively used in designing of white light emitting materials. Solid state lighting has a very bright future in various lighting applications because of their high efficiency and cost effectiveness compared to incandescent bulbs.

2. MATERIALS AND METHODS

For the synthesis of Sr₂CeO₄ doped with erbium solid state reaction method was used. The starting materials were strontium carbonate (SrCO₃), Cerium oxide (CeO₂), and Erbium oxide (Er₂O₃) were supplied by National Chemicals, Baroda (Gujarat State) of 99.9% purity. These materials were taken in Stoichiometric ratio of Sr : Ce as 2:1. SrCO₃ and CeO₂ with rare earth are weighed in molecular Stoichiometry. These all materials were ground in an agate mortar and pestle, grinded thoroughly to get fine powder [10-12]. This powder was taken in alumina crucible. After closing the cover the crucible was loaded in furnace and heated to the temperature 1200°C at the rate of 300°C/hr. The samples were kept at the set temperature for four hours then cooled down naturally. All the samples were prepared by same technique.

3. CHARACTERIZATIONS

The powder X-ray diffractograms (XRD) of the compounds were recorded using an automated Rigaku Miniflex X-ray diffractometer (D Max III VC, Japan). The observed (hkl) reflections and their intensities were compared with the calculated ones generated using the computer program POWD (an Interactive Powder Diffraction Data Interpretation and Indexing Program, Version 2.2). Photoluminescence excitation and emission spectra were recorded at room temperature using Spectrofluorophotometer (SHIMADZU, RF-5301 PC) equipped with a 150W Xenon lamp as excitation source. In this study, the morphologies of the phosphor powders were obtained by using the Nova NanoSEM450 [13-18]. Energy-dispersive X-ray spectroscopy (EDS, EDX, EDXS or XEDS), sometimes called energy dispersive X-ray analysis (EDXA) or energy dispersive X-ray microanalysis (EDXMA), is an analytical technique used for the elemental analysis or chemical characterization of a sample. The EXDA was recorded at Material Science department, M S University of Baroda.

4. RESULTS AND DISCUSSION

4.1 XRD Study

X-ray diffraction (XRD) is a versatile, non-destructive characterization method used for finding atomic and molecular structure of a crystal, crystal size, atomic parameters, stress measurement, etc. It works on the principle of diffraction. A crystal is a periodic array of atoms or molecules and hence it can act as scattering centers for X-rays. Electrons around the atoms or molecules are responsible for elastic scattering of the X-rays resulting in the diffraction. If X-ray falls on these periodic arrangements of atoms or molecules, destructive and constructive diffraction pattern obtained according to Bragg's Diffraction law: $2d_{hkl} \sin \theta = n\lambda$.

The structure and phase purity of the Sr_2CeO_4 doped Erbium the concentration (0.5 mol %) synthesized by solid state method was investigated by X-Ray diffraction method. Figure.1 XRD pattern of Sr_2CeO_4 : Er(0.5mol%) phosphor. The crystallite size of the particles of powder samples were calculated using Scherrer's equation $d = 0.9\lambda / \beta \cdot \cos\theta$, where β represents the full width at half maximum (FWHM) of XRD lines. The average crystallite size of the Sr_2CeO_4 phosphor is 22 nm. When Er doped with Sr_2CeO_4 the crystallite size is 35nm. The computer program POWD (an Interactive Powder Diffraction Data Interpretation and Indexing Program, Version 2.2) was used to calculate lattice parameters. The XRD patterns of the powders revealed that the structure of Sr_2CeO_4 is orthorhombic having lattice parameters $a=6.1200\text{\AA}$, $b=10.3600$, $c=3.5900$ and cell volume= $227.62(\text{\AA}^3)$, which agrees with the findings of previous research work of Danielson et al [1], Sankar et al [10].

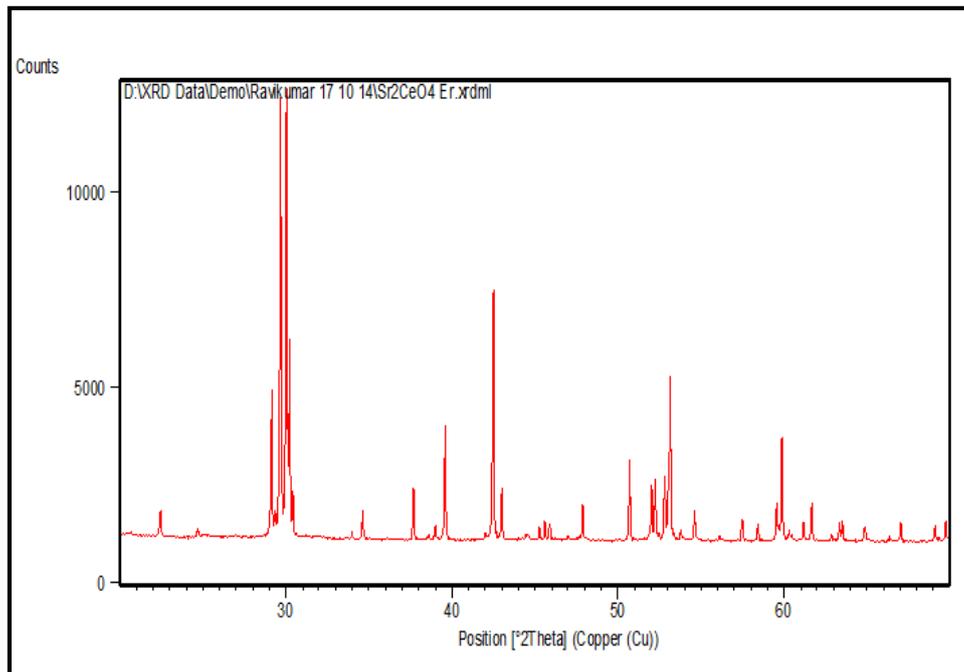


Figure 1 XRD pattern of Sr_2CeO_4 : Er (0.5mol %) phosphor

4.2. PL Study

The prepared Sr_2CeO_4 :Er³⁺(0.5 mol %) phosphors prepared by using solid state reaction method. The excitations are recorded by monitoring 470nm wavelength. All the phosphors are excited with 254, 275 and 340nm. Figure.1 shows the Excitation and Emission spectrum of Er(0.5mol%) doped Sr_2CeO_4 phosphor with different excitation wavelengths. When the phosphors are excited with various excitations, the basic Sr_2CeO_4 emission is found and the descending side i.e. 500 – 600nm contains the overlapped spiky emissions at 470, 527, 536, 545 and 558nm are clearly seen which the Er emissions are basically. It is interesting all the phosphors when excited with 275 and 340nm the basic emission around 470nm is out of range of instrument. The excitation spectrum of Sr_2CeO_4 shows two peaks around 254 nm and 340nm latter stronger than former these two excitation peaks may be related to different $\text{Ce}^{4+} - \text{O}^{2-}$ distances in the lattice [4,7]. Fig.2 shows that these two excitation peaks are attributed to the different charge transfer transitions. The excitation spectrum of Sr_2CeO_4 :Er³⁺ shows two peaks around 262nm and 362nm and former stronger than latter. The emission spectra for pure Sr_2CeO_4 when excitation wavelength is 262nm the emission peak is at 470nm shows broad band due to $f \rightarrow t_{ig}$ transition of Ce^{4+} perfect blue region with very good intensity [9]. However the effect of Er dopant modified the emission of Sr_2CeO_4 phosphor and the intensity was slightly decreased.

From the overall results of Er doped Sr_2CeO_4 phosphor it is concluded that the prepared phosphor can be used in devices like CFL and FL wherein 254nm excitation. It is also interesting the note the presence of Er in Sr_2CeO_4 gives raise many spiky peaks from 500 – 575nm greener component in the phosphor. This is due to the 3 un-paired electrons of Er and the associated resonance with 1 un-paired electron of Ce in the under study and their EN played an important role in resolving the green component of the phosphor [19-22]. From exciting the erbium doped Sr_2CeO_4 at $\lambda_{exc} = 262$ nm four transitions such as ${}^2\text{H}_{9/2} \rightarrow {}^4\text{I}_{15/2}$, ${}^4\text{F}_{3/2} \rightarrow {}^4\text{I}_{15/2}$, ${}^2\text{H}_{11/2} \rightarrow {}^4\text{I}_{15/2}$ and ${}^4\text{S}_{3/2} \rightarrow {}^4\text{I}_{15/2}$ was observed corresponding to the band position at 527, 536, 552 and 558 nm respectively. The ${}^2\text{H}_{9/2} \rightarrow {}^4\text{I}_{15/2}$ transition intensity is found to be high as compared to the other transitions when the concentration of Er³⁺ is 1.5 % in the host matrix. If concentration of Er³⁺ is gradually increased the intensity of ${}^2\text{H}_{9/2} \rightarrow {}^4\text{I}_{15/2}$ transition also gradually increased. The 527 and

536nm are assigned to ${}^2H_{11/2} \rightarrow {}^4I_{15/2}$ and 545 and 558nm are assigned to ${}^4S_{3/2} \rightarrow {}^4I_{15/2}$ are transitions of Er ion observed in the phosphor under study.

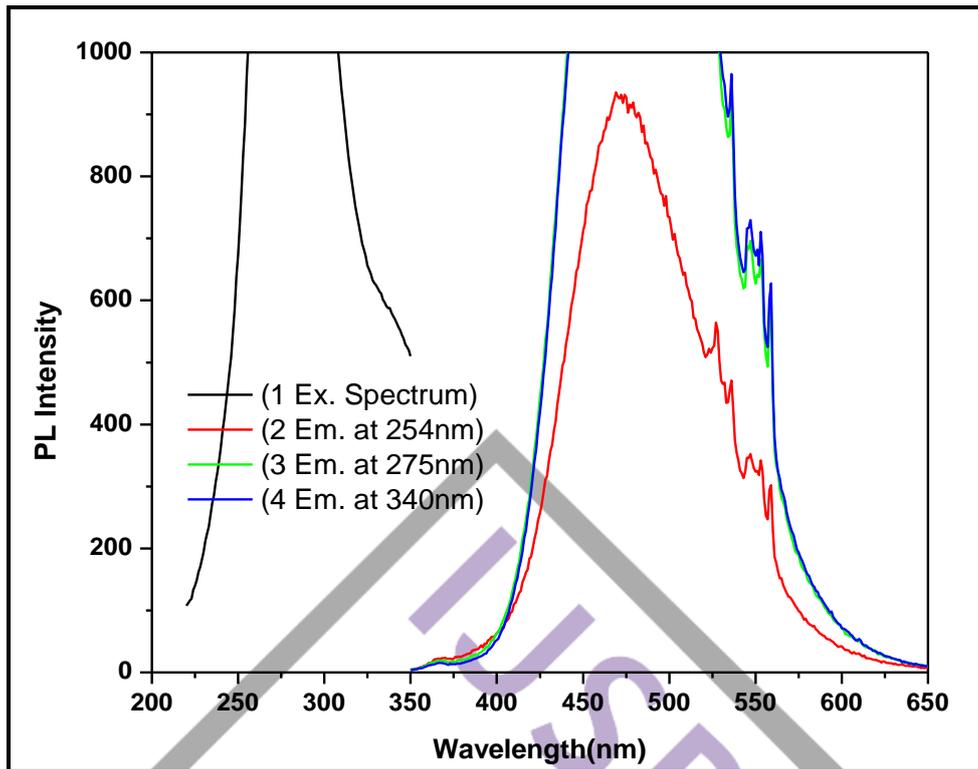


Figure.1 Excitation and Emission spectrum of Er(0.5mol%) doped Sr_2CeO_4 phosphor with different excitation wavelengths

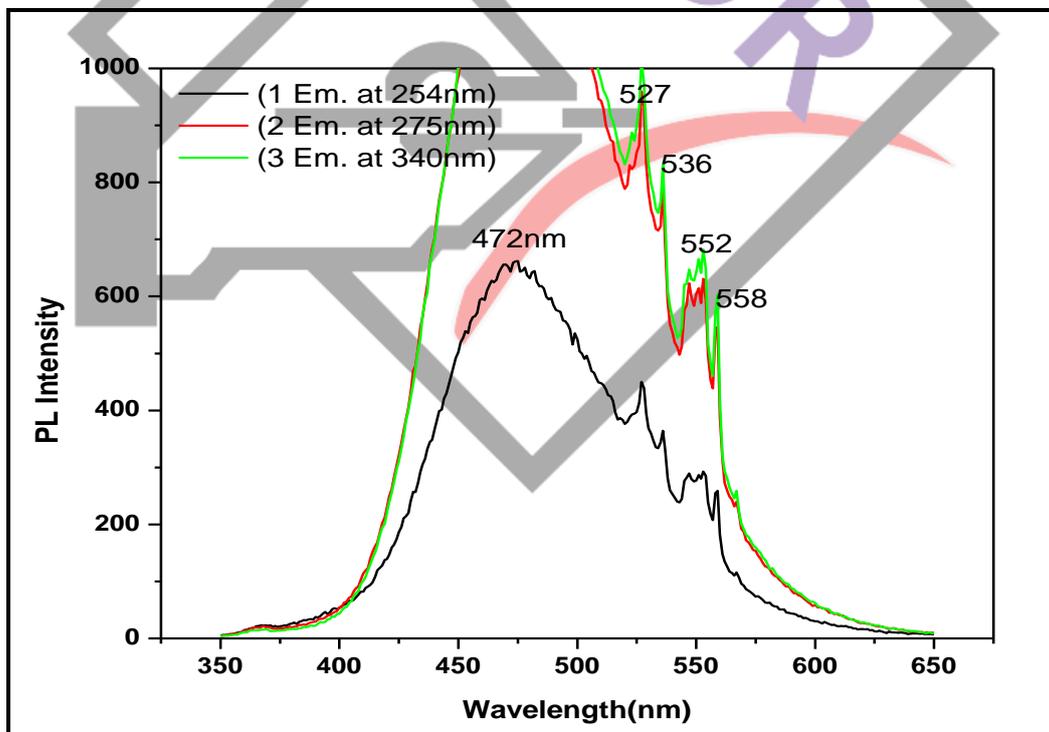
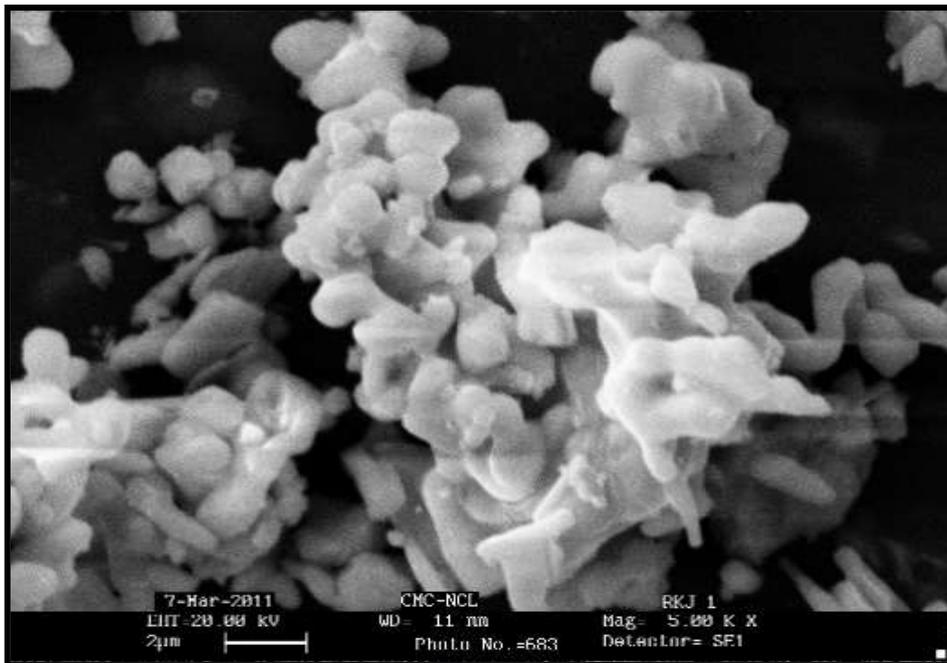
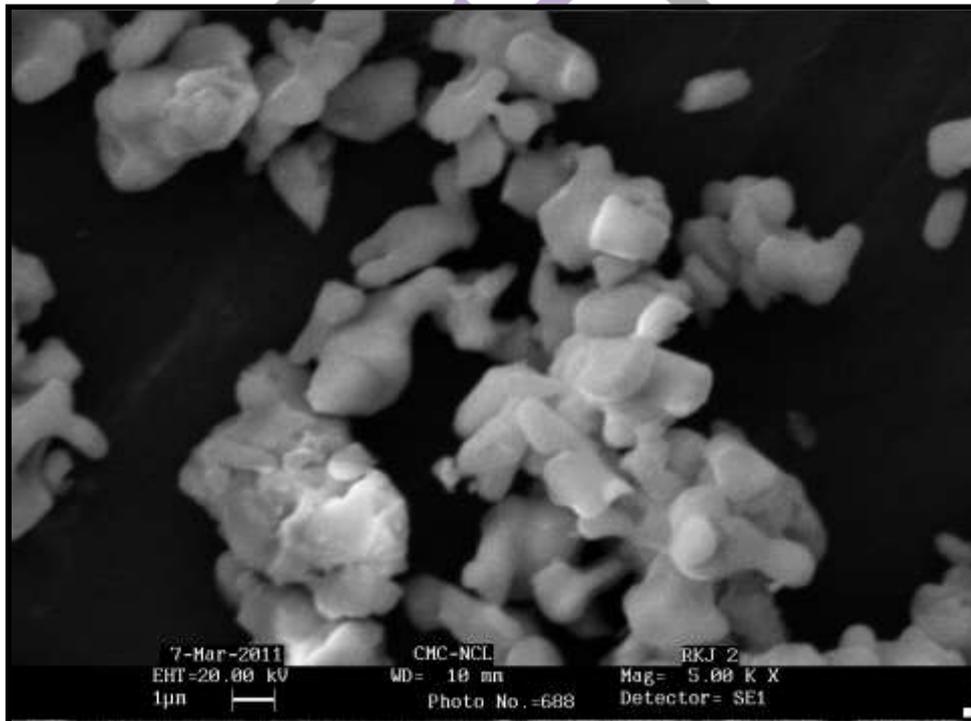


Figure.2 Excitation and Emission spectrum of Er(0.5mol%) doped Sr_2CeO_4 phosphor without different excitation wavelengths

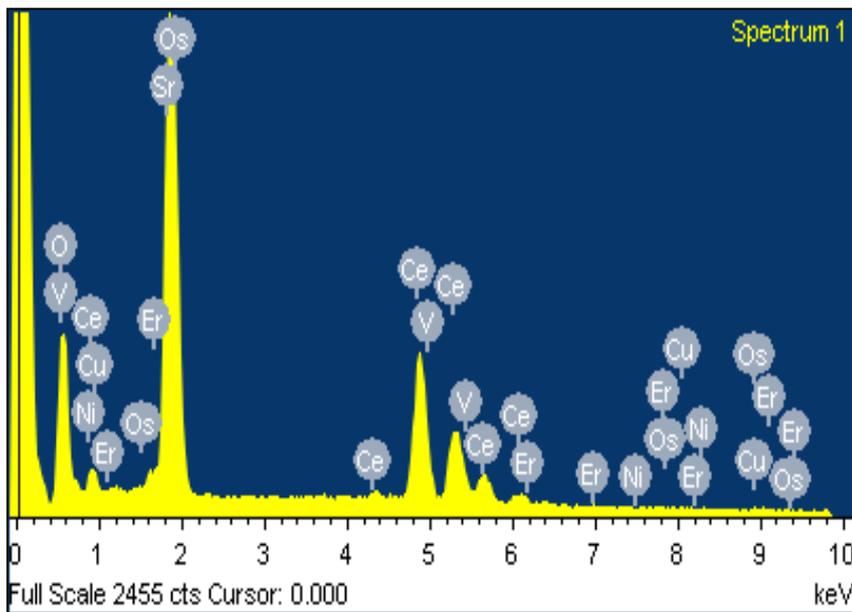
4.3 SEM Analysis

Fig.2 (a) Shows the SEM micrograph of pure Sr_2CeO_4 of and Fig. 2 (b) shows SEM micrograph of particles of $Sr_2CeO_4: Er$ (0.5mol%) phosphor which is appears to irregular shape having an average basal diameter of a few particles are loosely agglomerated. From SEM micrographs, it is found irregular shapes, agglomerated particles having Bessel sizes from 2- 6 microns are seen. The overall SEM studies concluding us the phosphors can be grounded mechanically and sieved to get uniform size to use as phosphors in the devices like Compact Fluorescent Lamps (CFL) and Fluorescent Lamps [FL].

Fig.2 (a) SEM micrograph of pure Sr_2CeO_4 PhosphorFig.2 (b) SEM micrograph of $\text{Sr}_2\text{CeO}_4:\text{Er}^{3+}$ (0.5mol %) Phosphor

4.4 EDAX Study

The prepared phosphors subjected to another characterization which is “Energy Dissipation through X-ray Elemental Analysis” (EDAX). Fig.3 EDAX data of $\text{Sr}_2\text{CeO}_4:\text{Er}$ (0.5%) phosphor of electron image, EDAX and the table containing of element, weight % and atomic % of the phosphors under study. From the figure of EDAX and the table basic phosphor elements are shown. It is concluded from the entire EDAX figure and table of dopant Er as well as Sr, Ce and Oxygen of various percentages are seen which are compared with the calculations made while preparing the phosphors. Therefore it is mainly concluded the formation of the phosphor is as per the empirical formula and weight percentage used to prepare phosphors using solid state reaction (SSR) method. It is also concluded the SSR method is to synthesize the phosphors under study is a very good method.



Element	Weight%	Atomic%
O K	19.66	62.37
V K	2.43	2.42
Ni K	-0.14	-0.12
Cu L	2.30	1.84
Sr L	31.66	18.34
Ce L	35.08	12.71
Er M	1.31	0.40
Os M	7.69	2.05
Totals	100.00	

Fig.3 EDAX data of $\text{Sr}_2\text{CeO}_4:\text{Er}(0.5\%)$ phosphor

5. CONCLUSIONS

- The XRD pattern confirms the formation of Sr_2CeO_4 compound in single phase and. The average grain size of the Sr_2CeO_4 phosphor is 22nm. And when Er doped with Sr_2CeO_4 the grain size is 35 nm.
- EDX spectra of $\text{Sr}_2\text{CeO}_4:\text{Er}$ in which the presence of Sr, Ce, O and Er are clearly identified.
- PL emission of pure Sr_2CeO_4 phosphor was observed at 470 nm which is blue emission this conform the formation of nano blue phosphor to good PL.
- $\text{Sr}_2\text{CeO}_4:\text{Er}^{3+}$ (0.5mol %) phosphor was observed when the excitation of the samples was kept at 262nm, the emission is in Bluish green color.
- The phosphor $\text{Sr}_2\text{CeO}_4:\text{Er}^{3+}$ (0.5mol %) shows good Photoluminescence, Intensity may be useful in various source lighting applications.

ACKNOWLEDGMENT

One of the authors (Ch. Atchyutha Rao) is grateful for the financial support from the University Grant Commission (UGC), New Delhi, India, under Minor research Project (MRP No: 4687/14-SERO/UGC).

And also thankful to the Principal and management of the Bapatla College of Arts & Sciences, Bapatla for Continues encouragement during this project work

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