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Preparation of nanosize$Sr_2SiO_4:Ce^{3+}$by oxalate precipitation method

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Abstract
In present work Sr2SiO4 sample activated by cerium ions were prepared first time by modified oxalate precipitation method after heating at 8000C for 2 hr. Sample are characterized by XRD, photoluminescence excitation, and photoluminescence emission. XRD pattern shows the formation of pure phase and nanosize powder. The emission spectra shows a broad peaks in the blue region (between 420 and 580 nm) , due splitting of the 4f energy level into the 2F5/2 and 2F7/2 energy levels of 4f1 electron in Ce3+ and influence by crystal field surrounding it in host material.
Keywords: nanomaterial, Sr2SiO4:Ce3+, photoluminescence, precipitation method, Dip-UV display application.

1. Introduction

Phosphors prepared by conventional methods are generally in micron size , but for solid state lighting purpose it should be small nanosized (<400 nm) and of uniform particle size, to reduce light scattering in the device to improving the extraction efficiency of the phosphors. Secondly it is not possible to grow crystals for LEDs that could give an emission at the desire wavelength. Hence it become a significant to prepare phosphors that could easily be synthesized by novel methods and could produce emission at desire wavelengths.

The synthesis of nanosize uniform powder by soft chemical methods is of particularinterest as they are simpler and do not need cumbersome equipment and machinery. This methods include precipitation under controlled condition, thus combustion method, Pechini sol-gel method, hydrothermal method and co-precipitation method are generally used for synthesis of oxide phosphors. In recent years , much attention has been focused onnanosize oxide based luminescent (specially for superior PL and CL properties) materials due to their commercial applications in color television, fluorescent tube, tri color LEDs etc [1,2]. The search for blue phosphors is of particular important because of limited number of stable blue luminescent materials available. The past twenty years have witnessed the development of AlGaN- based UV LEDs with their emitting wavelength shorter than 365 nm [3], (band gap of GaN). In 1998, Han et al.[4] manufactured the first UV LED with an emitting wavelength 353.6 nm. Subsequently, UV LED with emitting wavelength 280, 269 and 265 nm were achieved one by one [5-7] these UV LED can not be commercially utilized because of their lower quantum and luminous efficiency.

Recently developed (222-282 nm) AlGaN and InAlGaN based deep-LEDs on high – quality AIN template which can used as (long lifetime, high color – rendering light) for white light phosphor, and scientist Hirayama and his colleagues introduced an electron-blocking multi quantum barrier (MQB) into an AlGaN DUV-LED and achieved a dramatic increase in electron injection efficiency to more than 80% succeeding in generating light at a wavelength of 250 nm with a light output of 15 mW. These investigation will completely remove the use of mercury in lamp industries and fulfill the demand of second generation solid state lighting [8-9]. Thus after a decade it will possible that phosphorus with their exciting bands locating in UV region combined with UV LEDs to accomplish a desire white light.

From the literature serve it is found that Jong et al. [10], and QIAN Yanmin et al.[11] prepared Sr_2SiO_4 with rare earth ion doping

by solid state reaction at high temperature, in present work we proposed a new method to prepare Sr_2SiO_4 phosphor activated by cerium ions first time by precipitation method by sintering at 800^0C for 2 hr.

2. Experiment

Sr_2SiO_4phosphor activated with Ce^{3+}ions were synthesized by precipitation method shown in figure (1) in the form of flow chart. Using $Sr(NO_3)_2$ (99.99%), $Ce(NO_3)_3$(99.99%), SiO_2(99.99%) and Oxalic acid were used as starting material. First SiO_2 were dissolved in dilute HNO_3 (AR) to make nitrate solution and then $Sr(NO_3)_2$ and $Ce(NO_3)_3$ is added in silicate nitrate mixture. All sample were weighted with stoichiometric ratio. Then these starting material are heated till transparent solution is obtain at 80^0C near by half hour with continuous stirring. Solution of oxalic acid (dehydrated) was made in double distilled water and warming at 15-20 minute, with continuous stir till transparent solution is obtained. Then solution of oxalic acid was aided drop by drop in above mixture of nitrates .

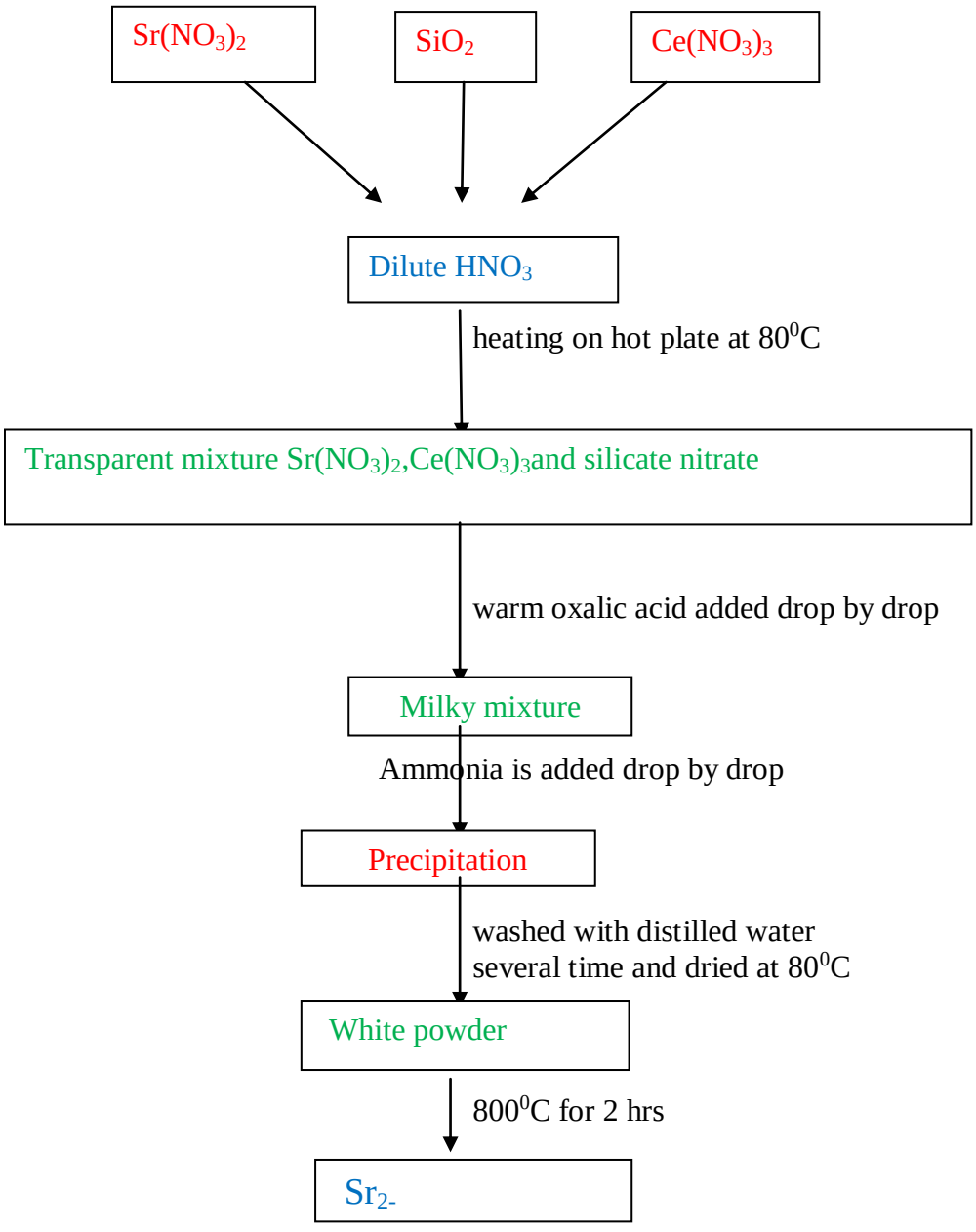

Fig.1. The flow chart for synthesis of $Sr_{2-x}SiO_4:Ce_x$ by precipitation method.

Then mixture keep well stir with adding ammonia till the precipitation is obtained. Then obtain precipitated was filtered out after cooling using double filter paper with washing with distilled water several times. Then it kept at 90^{0}C on hot plate for half hour for drying. The dry white powder was kept for heating at 800^0 C for 2h, to remove organic impurities.

3. Results & Discussion:

The XRD pattern of $Sr_2SiO:Ce^{3+}$phosphor with Ce^{3+} concentration 1 mol% is presented in fig.2. using Cu Kα radiation (λ= 1.541 A^o) with a nickel filter All patterns agree well with JCPDS file 39-1256, indicating that the doped Ce^{3+} ions have not caused any significant change in the host structure. Sr_2SiO_4 has an orthorhombic crystal structure, and their lattice parameters values are a = 0.7079nm, b = 0.5672nm, c = 0.9743 nm.

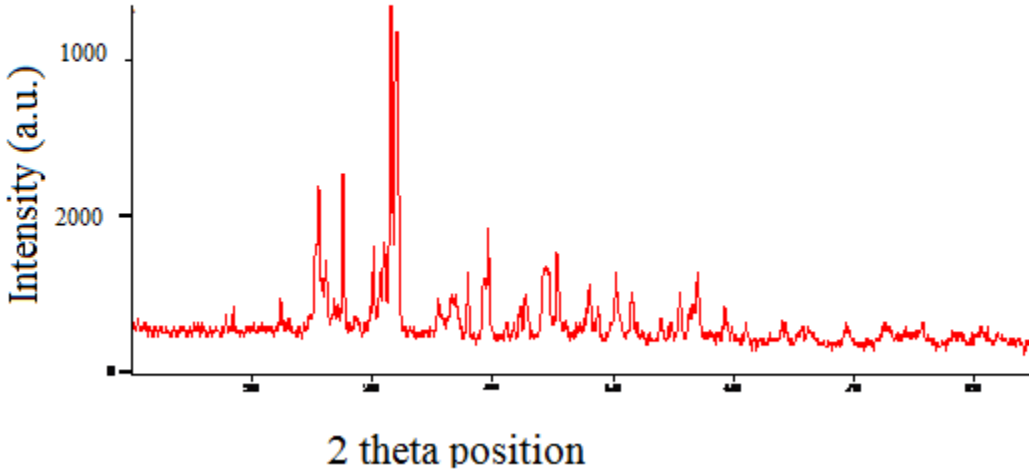

Fig.(2). XRD pattern of $Sr_2SiO_4:Ce^{3+}$

The crystalline size was calculated from the broad XRD peaks using the Scherrer equation (1).

$$d = \frac{0.9\lambda}{\beta \cos\theta} \qquad (2)$$

Where d is average grain size and β is the differacted full width at half maximum and λ is incident wavelength h, k, l are Millar indices and θ is Bragg's angle.The average crystalline size is found to be around 60 nm.

In the presence work $Sr_2SiO_4:Ce^{3+}$ have been studied in order to understand the The average crystalline size and luminescent behavior for application in the lighting industry and display applications. Figure (3) demonstrate the PLE spectra of $Sr_2SiO_4:Ce^{3+}$. Photoluminescence spectrum (λ_{em} = 470 nm) shows a broad band from 222 nm to 300 nm that is at deep- UV region.Figure (4) shows the photoluminescence spectrum, which shows a broad band in blue region of electromagnetic spectra.

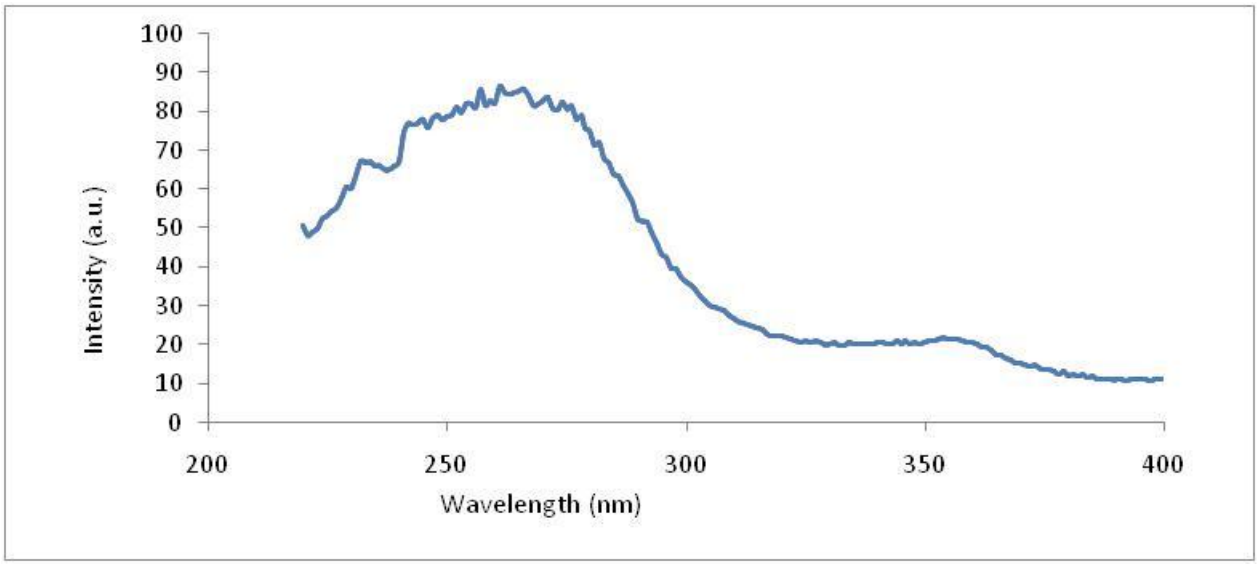

Fig.3. Excitation spectra of $Sr_2SiO_4:Ce^{3+}$ =1m% at emission wavelength 470 nm .

This luminescence in $Sr_2SiO_4:Ce^{3+}$ occurs due to characteristic transition (in the Ce^{3+} ion itself). When Sr_2SiO_4 doped with an activator such as Ce^{3+} creates an energy level structure inside the band gap of Sr_2SiO_4 ,where the 5d to 4f transition takes place. Splitting of the 4f energy level into the $^2F_{5/2}$ and $^2F_{7/2}$ energy levels is due to the $4f^1$ electron in Ce^{3+} having the ability to exhibit a +1/2 and -1/2 spin [12,13]. This creates the expectation of a luminescent spectrum with two main peaks in the blue region. However; reports on broad band single spectra (between

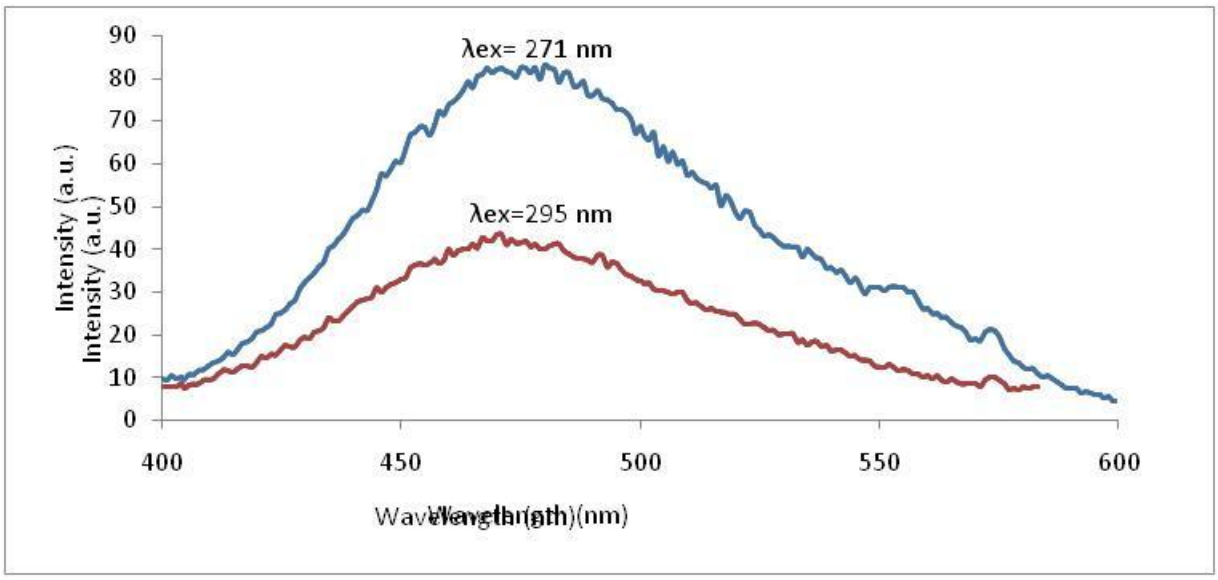

Fig.4. Emission spectra of $Sr_2SiO_4:Ce^{3+}$ =1m% .

420 and 580 nm) have been found, as shown in figure (3), this broad band in blue region indicates presence of strong crystal field on Ce^{3+} site in the proposed host material. Since Sr_2SiO_4 exhibit excellent physical and chemical stability. Besides, it absorbed ultraviolet radiations and emit the blue light when activated by cerium ions thus it may play positive in display applications.

4. Conclusion:

An simple and effective precipitation method is was used to preparenanosize and single phasedSr_2SiO_4 activated by cerium ions. Average crystalline size is found to be around 60nm. Phosphors showed a intense broad band emission in the blue region when it was excited with deep ultraviolet radiations. Presence of broad band indicates, existence of strong crystal field on Ce^{3+} site in Sr_2SiO_4 :Ce^{3+}. Thus proposed phosphors can be used for display applications under deep-UV LED excitations.

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