



# INFLUENCE OF FLUX ON THERMO LUMINESCENCE PROPERTIES OF $Sr_2SiO_4: Eu_{0.01}$ NANOPHOSPHOR

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## ABSTRACT

$Eu^{3+}$  doped (0.01 mol %) nanocrystalline  $Sr_2SiO_4$  phosphor was prepared by solution combustion method using citric acid as fuel and NaF as flux. The crystallization and particle size of  $Sr_2SiO_4$  have been investigated by using Powder X-ray diffraction. Thermoluminescence of  $Sr_2SiO_4: Eu_{0.01}$  nanophosphor exposed to  $\gamma$ -irradiation in the dose range 10-100 Gy has been investigated. Two glow peaks recorded at 156°C and 220°C respectively. The kinetic order, activation energy and frequency factor were estimated by Chen's peak shape method.

**Keywords:** Combustion synthesis, Nanocrystals, Thermo luminescence.

## INTRODUCTION

Thermoluminescence (TL) is an important tool for estimating the luminescent center in solids. Stimulated emission of light from an insulator or semiconductor after heating following the previous absorption of energy from ionizing radiation is known as TL (1). Recently TL materials find application in so many fields. The important application of TL materials is in TL dosimetry (2).

In recent years silicates have attracted the research community much because of their structure, properties like physical, chemical, thermal stability etc and it's easy to implant other ions into the host lattice. So we have selected Eu as a dopant (3,4).

In phosphors, the emission efficiency depends on size and its distribution. It's difficult to have a control on the size of the particle during synthesis which can be solved by using flux. A flux is an additive which does not affect the reactants during the calcination, instead it promotes the crystal growth of the final product. The addition of flux has great influence on the particle size, ion diffusion, and crystallization process. This also decreases the activation energy of the reaction by forming a thin film of liquid around the particles which contributes for the betterment of the morphology (5,6).

Li Zhang et al (7) have studied the TL properties of  $Pr^{3+}$  doped  $Sr_2SiO_4$  and showed that the phosphor materials is long lasting one. Lakshminarasimhan and Vardaraj (8) reported the TL studies of rare earth doped  $Sr_2SiO_4:Eu_{0.01}$ . Literature survey also reveals that addition of fluxes into various phosphors will improve optical



properties of phosphor. Hai Guo et al have synthesized  $\text{Sr}_2\text{SiO}_4:\text{Eu}$  yellow phosphor using different fluxes and showed  $\text{NH}_4\text{F}$  is superior (9). Jee Hee Lee et al have prepared  $\text{Sr}_2\text{SiO}_4:\text{Eu}$  by ball milling method using  $\text{NH}_4\text{Cl}$  as flux and studied the correlation between phase transition and luminescence properties (10).

With the development of technology several synthesis methods are available for the synthesis of  $\text{Sr}_2\text{SiO}_4:\text{Eu}_{0.01}$  such as solid state, sol-gel, spray pyrolysis, etc. However there are some disadvantages in these methods like process is complex, expensive, long duration etc where as combustion method has so many advantages over other methods. It produces homogeneous, nanoparticles within a short duration of time (11, 12). In this work combustion synthesis was used to prepare  $\text{Sr}_2\text{SiO}_4:\text{Eu}_{0.01}$  phosphor using citric acid as fuel and  $\text{NaF}$  as flux. The influence of flux on the TL properties has been studied.

## II. EXPERIMENTAL

### 2.1. Materials and Procedures

The starting materials were strontium nitrate ( $\text{Sr}(\text{NO}_3)_2$ ), europium oxide ( $\text{Eu}_2\text{O}_3$ ), fumed silica ( $\text{SiO}_2$ ), sodium fluoride ( $\text{NaF}$ ), citric acid ( $\text{C}_6\text{H}_8\text{O}_7$ ).  $\text{Eu}_2\text{O}_3$  was converted into nitrate by dissolving in 1:1  $\text{HNO}_3$ . The stoichiometric amounts of starting materials were dissolved in a minimum amount of water to obtain a homogeneous solution and then stirred. After that the mixture solution were introduced into a muffle furnace maintained at temperature of  $500^\circ\text{C}$ . Initially the solution boiled and underwent dehydration followed by decomposition with escape of large amount of gases and then spontaneous ignition occurred and underwent combustion. The whole process is over with in 5 min. after combustion the product was calcined at  $1100^\circ\text{C}$  for 3hrs.

### 2.2. Experimental Tests

The synthesized powder was characterized by PXRD, it was carried out using Philips and Siemen's D5005 X-ray diffractometer using  $\text{Cu K}\alpha$  ( $\lambda=1.5418\text{\AA}$ ) radiation with a nickel filter. FTIR spectra were recorded with a Perkin Elmer FTIR spectrometer. TL glow curves were recorded for  $\gamma$  - irradiated powder sample at room temperature using Nucleonix TL reader.

## III. RESULTS AND DISCUSSION

### 3.1. Powder X-Ray Diffraction (PXRD)

The XRD pattern of  $\text{Sr}_2\text{SiO}_4:\text{Eu}_{0.01}$  phosphors are shown in figure 1. The observed diffraction patterns of  $\text{Sr}_2\text{SiO}_4:\text{Eu}_{0.01}$  nanophosphors are well matched with  $\alpha$ -phase (monoclinic) of  $\text{Sr}_2\text{SiO}_4$  with JCPDS file no 39-1256. Further it is observed that the intensity of the XRD peaks increases with the addition of flux, the particle size was estimated using Scherrer's method are found to be in the range 20-30nm for the one prepared without flux and 30-40 nm for the one with flux.

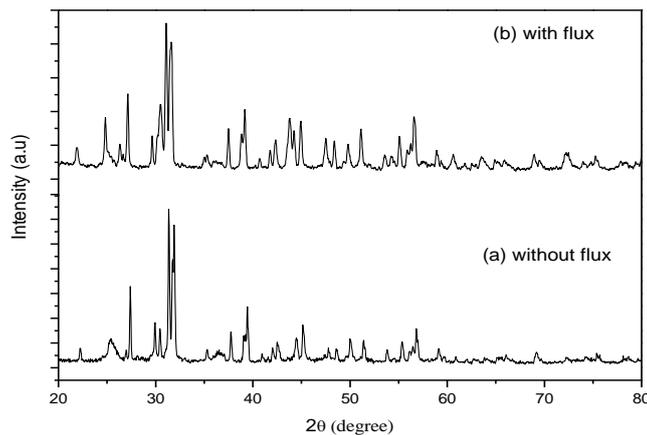


Fig 1: PXRD of  $\text{Sr}_2\text{SiO}_4: \text{Eu}_{0.01}$  (a) without flux (b) with flux.

### 3.2. Fourier Transform infrared Spectroscopy

FTIR was employed as an auxiliary characterization alternative. Fig 2 displays the spectra of  $\text{Sr}_2\text{SiO}_4: \text{Eu}$  without and with flux. The existence of characteristic Sr-O stretching and Si-O stretching confirmed the structure. Changes in the shape of peaks were observed with and without flux. The vibrations corresponding to Sr-O and Si-O are mentioned here 498–  $\text{F}_2$  bending ( $\gamma_4$ ), 503– symmetric stretching vibration of Si-O-Si, 726– 1073– asymmetric Si-O-Si bond, Si-O  $\text{F}_2$  ( $\gamma_3$ ) stretching, 1480 ( $\text{Cm}^{-1}$ ) – Sr-O stretching vibrations.

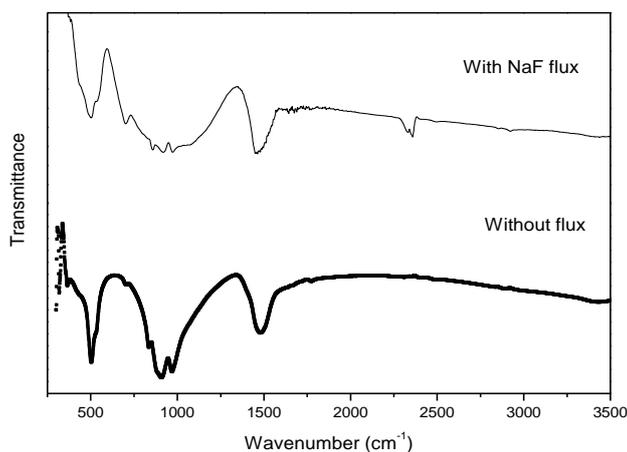
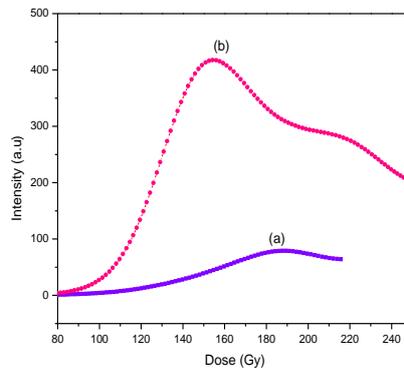


Fig 2: FTIR of  $\text{Sr}_2\text{SiO}_4: \text{Eu}_{0.01}$  (a) without flux (b) with flux.

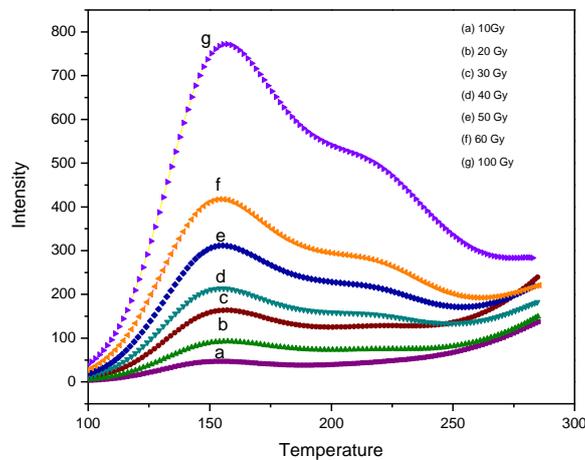
**3.3. Thermoluminescence (TL)**

TL curves of  $Sr_2SiO_4: Eu_{0.01}$  phosphor with and without flux exposed to 10 Gy  $\gamma$  irradiation i.e curve ‘a’ and ‘b’ are shown in figure 3. It is obvious that the two curves are very different in shape, peak position and TL intensity. Curve ‘a’ consist of only one broad peak at 189°C where as curve ‘b’ has the main TL peak at about 156°C and another weak shoulder at 220°C. The peaks in the TL curve are related to two different traps present in the material. The difference in the TL curves shows that the flux has a subsequent effect on the traps of the material.



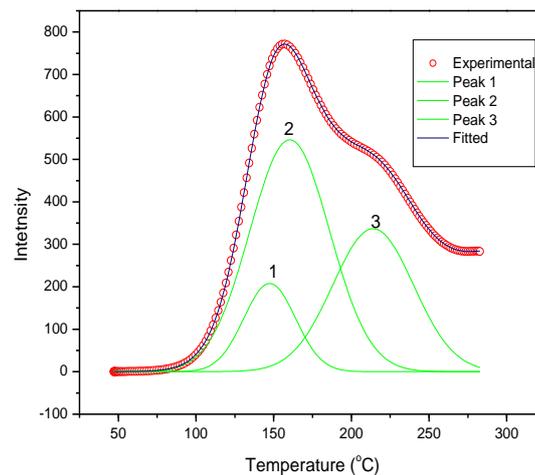
**Fig 3: TL glow curves of  $Sr_2SiO_4: Eu$  (a) without flux (b) with flux for 10Gy  $\gamma$ -irradiation**

TL glow curves of  $Sr_2SiO_4: Eu_{0.01}$  with flux for different doses (10-100 Gy) under  $\gamma$ -irradiation is shown in figure 3. It is observed that the intensity increases with increase in dose, the shape and position of the TL peaks remains almost constant in the studied dose range.



**Fig 4: TL glow curves of  $Sr_2SiO_4: Eu_{0.01}$  phosphor with NaF flux for different dose range**

The experimentally obtained curve for 100 Gy was deconvoluted to calculate the kinetic parameters using Chen’s formulae (13) and results are summarized in Table 1.



**Fig 4: Deconvoluted TL Glow curve of Sr<sub>2</sub>SiO<sub>4</sub>: Eu<sub>0.01</sub> Phosphor with NaF flux**

**Table 1: Kinetic parameters estimated from Chen’s method**

Peak	$\mu_g$	E (ev)	T <sub>m</sub> (°C)	S (s <sup>-1</sup> )
1	0.49	1.16	147.3	1.72X0 <sup>15</sup>
2	0.5	0.8	160	2.68X10 <sup>10</sup>
3	0.49	0.99	214	2.27X10 <sup>11</sup>

### CONCLUSIONS

Sr<sub>2</sub>SiO<sub>4</sub>: Eu<sub>0.01</sub> phosphor was by solution combustion method with and without flux.

1. It's observed that particle size increases with the addition of flux.
2. Further TL analysis suggests the presence of single and two traps for the phosphor with and without flux respectively.
3. The addition of flux has significant effect on the defect structure of the material.
4. Intensity increases with the addition of flux even though the particle size is more. It's because of sensitivity. Larger particles are more sensitive towards irradiation. Thus our sample can be used as a good dosimetric sample.

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