

Thermoluminescence of nanocrystalline Eu doped BaSO₄

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Abstract: Nanocrystalline powder of 0.1 mol% Eu doped BaSO₄ were synthesized by chemical co-precipitation technique. The formation of the powder was confirmed by the Powder X-ray Diffraction (PXRD) analysis. Shape and size of the synthesized powder were examined using Transmission Electron Microscope (TEM). Dosimetric characteristics have been carried out of the γ -ray irradiated BaSO₄:Eu phosphors using Thermoluminescence (TL) technique. The TL glow curve of the samples shows the dosimetric peak at around 456 K which is sufficient above the room temperature. Composite TL glow curve of the synthesized powder deconvoluted using general order kinetics equation and also calculated the kinetic parameters.

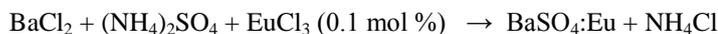
I. Introduction

High TL sensitive BaSO₄ is one of the most TLD phosphor used in radiation dosimetry using thermoluminescence technique [1]. The various activators such as Mn, Cu, Eu etc are used for the better TL sensitivity and more stability [2]. It used as TLD for personal and environmental dosimetry due to its high TL sensitivity, chemically and thermally stability and low cost availability. Currently, nanotechnology and nanomaterials have attracted several researchers from different fields [3], especially from the field of luminescence. It has been found that the physical properties of individual nanoparticles can be very different from those of their bulk counterparts. Recent studies on different luminescent nanomaterials have showed that they have a potential application in dosimetry of ionizing radiations for the measurements of high doses using the TL technique, where the conventional microcrystalline phosphors saturate [4–8].

In the present study we synthesized the sample via chemical co-precipitation route and analyzed the TL characteristics of nanocrystalline Eu doped BaSO₄ for the dosimetry purpose. The kinetic parameters were also calculated to get the proper information about the trap centers within the forbidden gape of the materials.

Experimental

Nanocrystalline powder of BaSO₄ was synthesized by Chemical co-precipitation route. The starting materials were BaCl₂·(NH₄)₂SO₄ and the EuCl₃ as dopant were taken of analytic grade (AR). The samples were prepared stoichiometric taking into consideration the following chemical reaction:



The appropriate amount of BaCl₂ was dissolved in the triply distilled water to prepare the aqueous solution and impurity salt also adding into this solution then stirring on the magnetic stirrer. The solution of (NH₄)₂SO₄ was then added into the above solution stoichiometrically drop wise in the presence of ethanol. Whole the solution was stirred for 3 hours for the completion the reaction. White precipitate of the prepared sample separate from the solution by centrifuging at 5000 rpm and precipitate washed several times with triply deionized water. The collected precipitates were dried at 373 K for 12h in the oven. For the further characteristics the sample was annealing at 500 °C for 30 min in the programmable oven in the nitrogen atmosphere.

Instrument Used For Characterization

The Powder X-Ray Diffraction (PXRD) patterns were recorded using a high-resolution D8 Discover Bruker X-ray Diffractometer. The surface morphology of the samples were observed using high resolution images were taken on Philips Tecnai G2 30 Transmission Electron Microscope (TEM). Thermoluminescence (TL) was recorded on Harshaw TLD Reader 3500 HT. The TL of the samples was taken immediately after the irradiation. For recording TL, 5 mg of the pre-irradiated sample to γ -rays from the ¹³⁷Cs source to different doses in the range (0.1Gy–100 Gy) at room temperature (RT) was taken every time and heated linearly with the heating rate 5K/Sec.

II. Results And Discussion

1.1 PXRD and morphology of synthesize powder.

Fig. 1 shows the PXRD pattern of nanocrystalline BaSO₄:Eu phosphor at room temperature. The XRD pattern of Eu doped BaSO₄ samples were found in good agreement with the reported orthorhombic structure with space group Pmna53 data of barium sulfate having JCPDS card no: 00-001-1229 [9].

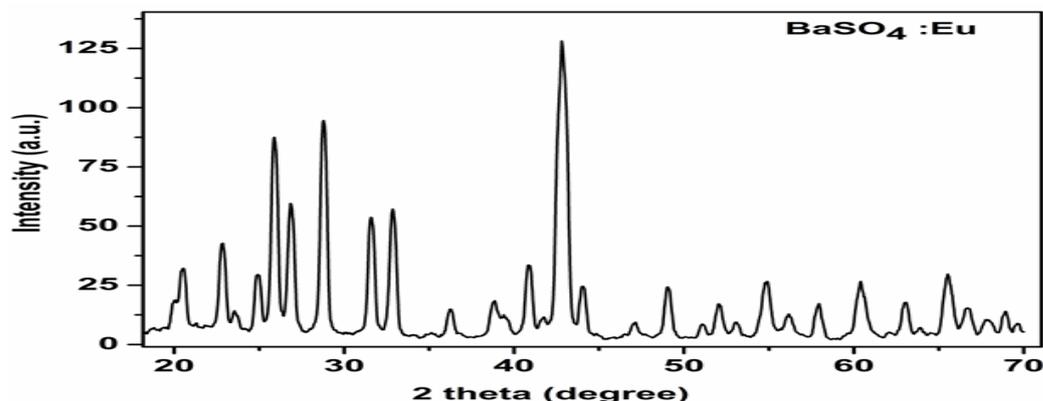


Figure 1: PXRD pattern of nanocrystalline BaSO₄:Eu powder

The morphology of the synthesize nanocrystalline powders were examined by TEM micrograph as shown in Fig. 2. The diffraction pattern of the electron beam from the atom site/ lattice points of plane shown in inset of the Fig. 2 which agrees the crystalline nature of the prepared materials. The irregular shaped particles of the materials could be seen in the both micrographs. The average particles size from the TEM micrograph was observed 60- 100nm. EtOH play the key role to degrade the particle size of the prepared materials by the asymmetric chains of the hydrogen bonding formed during the synthesis due to disproportionate ratio of water and EtOH [8].

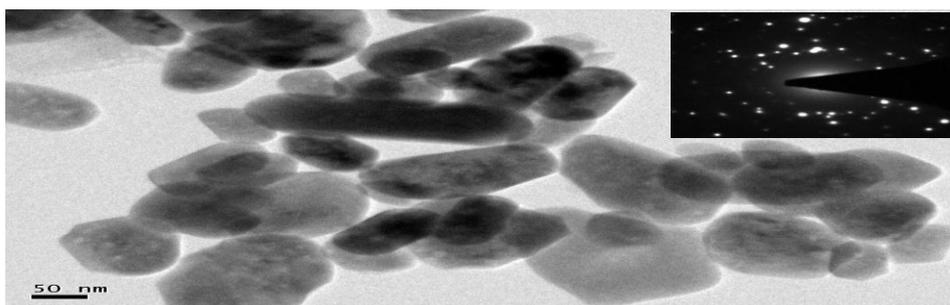


Figure 2: TEM image of nanocrystalline BaSO₄:Eu powder

1.2 Thermoluminescence (TL) properties

The TL glow curve of the pre annealed γ -ray exposed (0.1- 100 Gy) samples of synthesized nanocrystalline BaSO₄:Eu shown in fig. 3. It was found a single dosimetric peak at around 456 K from the TL glow curve which is sufficient above the room temperature. Numan Salah and the co-workers [10] reported two dosimetric peak at 462 K and other as a shoulder at 497 K in nanocrystalline BaSO₄:Eu. A small peak shift may be attributed to the change in particles shape and size [8]. In radiation dose testing, the synthesized sample was shown the linear dose response as shown in fig 4.

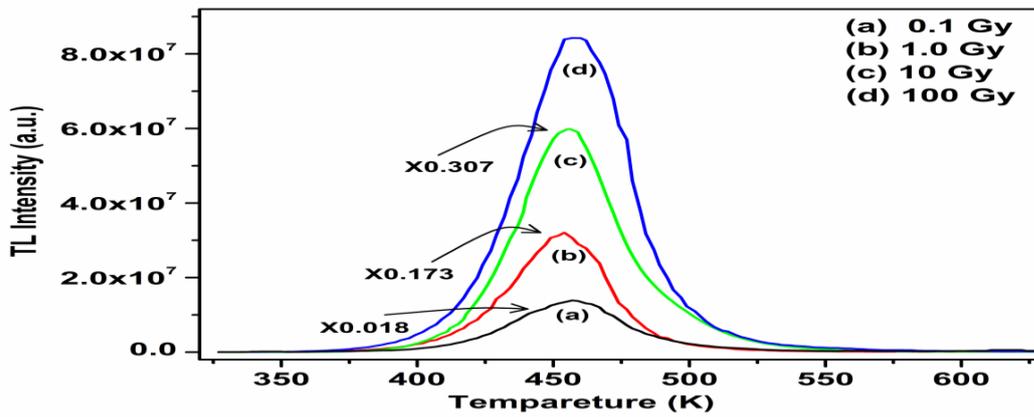


Figure 3: TL glow curves of nanocrystalline BaSO₄:Eu powder exposed to various doses of γ -rays from a ¹³⁷Cs source. The ordinate is to be multiplied by an integer near the glow curve to get the relative intensity.

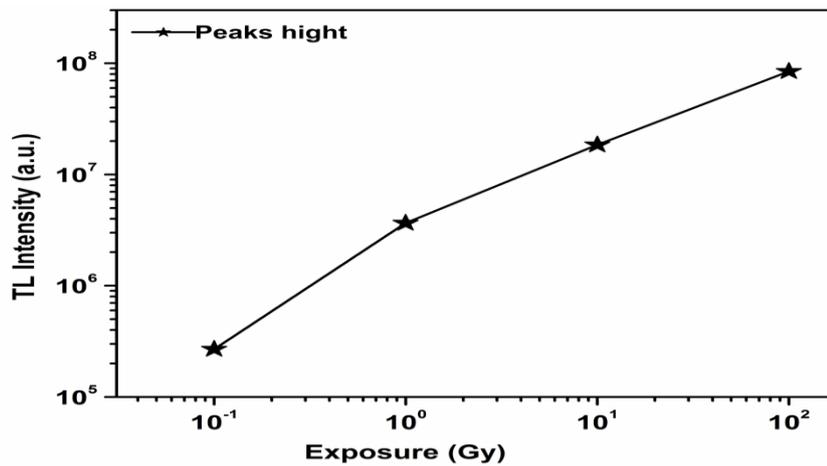


Figure 4: TL dose response of nanocrystalline BaSO₄:Eu phosphor.

The recorded TL glow curve has been deconvoluted by a computerized program, Glow-Fit [11]. The glow curve of nanocrystalline powder BaSO₄:Eu of two deconvoluted peaks shown in the Fig. 5. The quality of best fitting describing by a parameter is called the Figure of Merit (FOM), calculated as the given relation

$$FOM(\%) = \frac{\sum_i |y_i - y(x_i)|}{\sum_i y_i} \times 100\%$$

Where y_i is the content of the channel i and $y(x_i)$ is the value of the fitting function in the center of channel i . If the Glow curves with FOM values excess of 2.5% it means further investigation to determine the reasons for the poor fit. In this work, fitting of the composite glow curve of MgF₂:Mn²⁺ deconvoluted by 2 isolated peaks with the best value of FOM 1.18%.

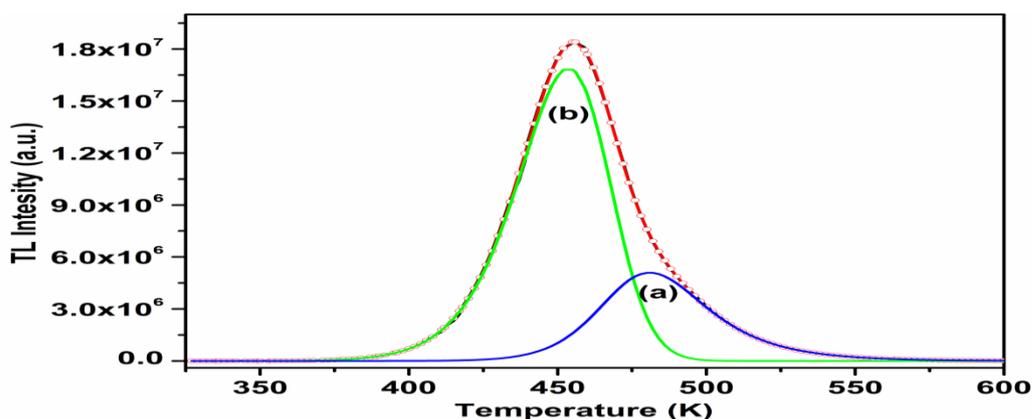


Figure 5. Deconvolution TL glow curve of BaSO₄:Eu phosphor exposed at 10 Gy of ¹³⁷Cs source.

Table 1. Kinetic parameters of the deconvoluted TL glow curve of nanocrystalline BaSO₄:Eu

Sample	Peak	Peak Temp.(K)	FOM (%)	E (eV)	Kinetic order (b)	Frequency factor (S)
BaSO ₄ :Eu	a	453	1.18	1.22	1.19	1.32X 10 ¹³
	b	481		1.75	2.21	8.87X 10 ¹⁷

The values of the kinetic parameters of all isolated peaks of the glow curves at the heating rate $\beta = 5$ K/s were calculated by the glow curve deconvolution (GCD) method, they are given in the Table 1. Kinetic parameters give the proper information about the trap centers and stored energy after irradiation of the sample.

III. Conclusion

Nanoparticles of Eu doped BaSO₄ were successfully synthesized by the chemical co-precipitation methods. The average particles size 80 nm were controlled by taking the appropriate concentration of starting materials and EtOH ratio. The intense dosimetric peak was found at around 456 K, which shows linear dose response. Some favorable characteristics as non-hygroscopic, high TL sensitivity, chemically and thermal stability of nanocrystalline Eu doped BaSO₄ makes it suitable phosphor for the radiation dosimetry.

IV. Acknowledgement

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