

## THERMOLUMINESCENCE STUDIES IN COMBUSTION

SYNTHESIZED  $\text{Mg}_2\text{SiO}_4:\text{Dy}^{3+}$  NANOPHOSPHOR

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*Bangalore University, Jnanabharathi campus, Bangalore-560 056***ABSTRACT**

Nanocrystalline porous  $\text{Mg}_2\text{SiO}_4$  also known as 'Forsterite' doped with  $\text{Dy}^{3+}$  has been synthesized by a novel low temperature initiated self propagating gas producing solution combustion process using urea as a fuel. The phosphor has been characterized using XRD and FTIR techniques and the particle size is measured to be 40-60nm. Thermoluminescence of  $\text{Mg}_2\text{SiO}_4:\text{Dy}^{3+}$  gamma irradiated for doses in the range 2.192kGy to 5.822 kGy have been studied at room temperature. A prominent glow with peak at 160°C besides three weak and unresolved glows is observed and they are tentatively attributed due to recombination of F center electrons with holes associated with  $\text{SiO}_4$ ,  $\text{Mg}^{2+}$  and due to f-f transitions of  $\text{Dy}^{3+}$  ions. It is observed that TL intensity increases with increase in  $\gamma$ -ray dose. The TL glow curves are analyzed using Chen's glow curve shape method and the results obtained are discussed here.

**INTRODUCTION**

Nanomaterials have attracted considerable attention because of their unique chemical, physical, electrical, magnetic, optical and mechanical properties. Because of these properties, they are useful as catalyst, sensors, coating materials, tunable lasers, and memory devices. Thermoluminescence also known thermally stimulated luminescence is a type of delayed phosphorescence, where the photon is released when the crystalline substance is heated from low temperature to high temperature. The release of the trapped charge carriers by thermal stimulation and their subsequent recombination with their counter parts produces the light called thermoluminescence [1-6]. Thermoluminescence is extensively used in radiation dosimetry, Forensic science, for monitoring integrated radiation exposure, archeology and geological dating [7]. The study of thermoluminescence in materials reveals the nature and extent of radiation damage caused to the material. TL technique is successfully used to study the dynamics of electrons and holes associated with point defects such as color centers [8, 9].

In the present work, a simple and novel method is used to synthesize nanocrystalline Forsterite ( $\text{Mg}_2\text{SiO}_4$ ) by a low temperature initiated solution combustion (LCS) route using urea as a fuel and the corresponding metal nitrates as oxidizers. Instantaneous combustion synthesis is an important powder processing technique generally used to prepare nanocrystalline oxide ceramics with homogeneous and high surface area. The technique involves several advantages like fast heating rates, short reaction time (~5 min), and economical [10].

**EXPERIMENTAL**

$\text{Mg}_2\text{SiO}_4:\text{Dy}^{3+}$  nano powders used in this experiment is synthesized through LCS method based on procedure discussed elsewhere [11]. Synthesized powder is grained into a fine powder using an agate mortar and calcined at 800°C for three hrs. The sample is characterized by PXRD (Philips PW 1050/70, Cu  $K\alpha$  radiation with Ni Filter). The sample is irradiated with gamma rays ( $\text{Co}^{60}$ ) for the dose ranging from 2.912 to 5.822kGy. Thermoluminescence glow curves are recorded with a home made TL set up consisting of a small metal strip heating directly using a temperature programmer, photomultiplier (931A) and a multimeter (Rishicom) at a heating rate of  $5.5^\circ \text{C s}^{-1}$ .

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## RESULTS AND DISCUSSION

The phase purity and crystal structure are examined by PXRD using Cu  $K_{\alpha}$  radiation of wavelength 1.54056 Å. Figure 1 shows the PXRD patterns of pure and doped samples synthesized via combustion technique. The XRD pattern is found to match exactly with those reported in the literature [12]. The average particle size (D) is estimated from the line broadening in X-ray powder using Scherrer's formula [ $D = (K\lambda)/\beta \cos\theta$ ], where, 'K' is a constant, ' $\lambda$ ' wavelength of X-rays, and ' $\beta$ ' FWHM] is found to be 40-60nm [13].

Figure 2 shows the thermoluminescence glow curves of  $Dy^{3+}$  doped magnesium silicate  $\gamma$ -rayed for doses in the range 2.912 to 5.822kGy. The glow curves clearly show a well-resolved glow with peak  $\sim 160^{\circ}C$  ( $T_{g1}$ ) and shoulders with peaks at 103, 234 and  $266^{\circ}C$ . The glow peak temperature of  $T_{g1}$  is almost steady for the entire dose range and the variation of TL peak intensity with gamma ray dose is shown in Figure 3. The peaks around 160 and  $234^{\circ}C$  are likely arise from recombination of charges released from  $F^+$  center near  $Mg^{2+} / Dy^{3+}$  sites [14] and peak around  $266^{\circ}C$  may tentatively be attributed due to recombination of F center electrons with holes associated with  $SiO_4$  and  $Mg^{2+}$  sites. The kinetic parameters are calculated according to glow curve shape method (modified by Chen) using computerized glow curve deconvolution technique [15-16] and are shown in Figure 4 and the results obtained are tabulated in Table1. The dominant trapping centers of a phosphor with trap depths in the range 0.7 to 1.1 eV shows excellent phosphorescence property and it may be responsible for enhanced afterglow emission [17].

## CONCLUSIONS

$Mg_2SiO_4:Dy^{3+}$  nanocrystalline phosphor has been prepared at very low temperature and in a very short time using solution combustion technique. The PXRD pattern showed the presence of alpha phase and the particle size calculated using Scherrer's formula confirms the nano size. The phosphor exhibits good TL response and it is suggested that the phosphor may be used as a candidate for high radiation dosimeter.

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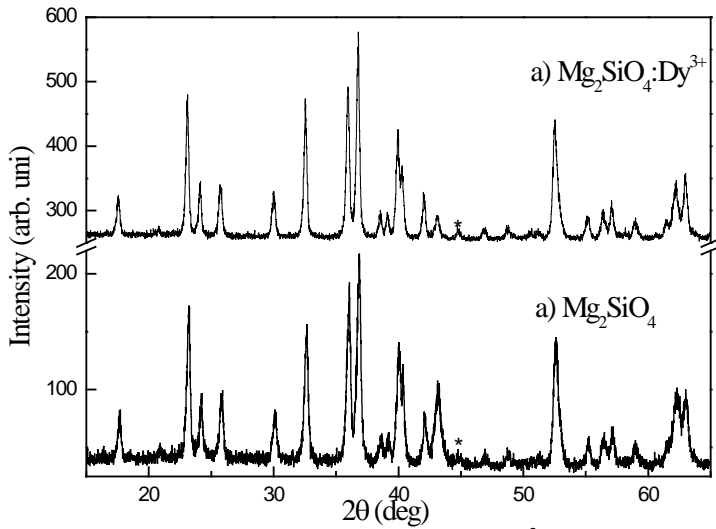


Figure 1. PXRD Patterns of (a) pure and (b) Dy<sup>3+</sup> doped Mg<sub>2</sub>SiO<sub>4</sub>.

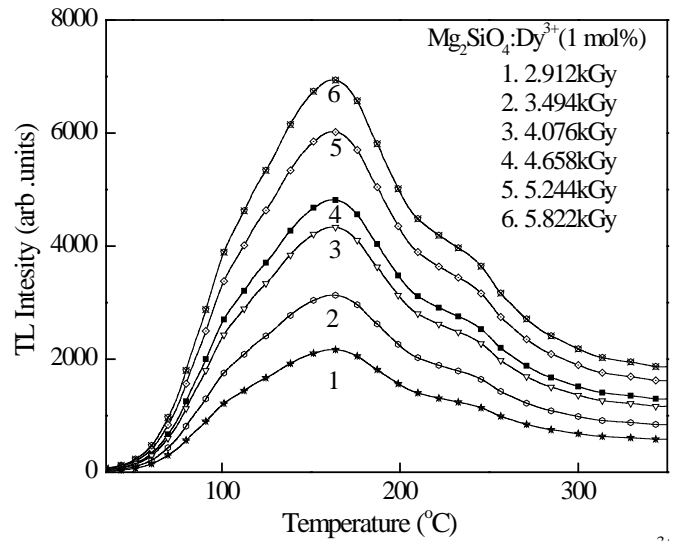


Figure 2. Thermoluminescence glow curves of Mg<sub>2</sub>SiO<sub>4</sub>:Dy<sup>3+</sup>

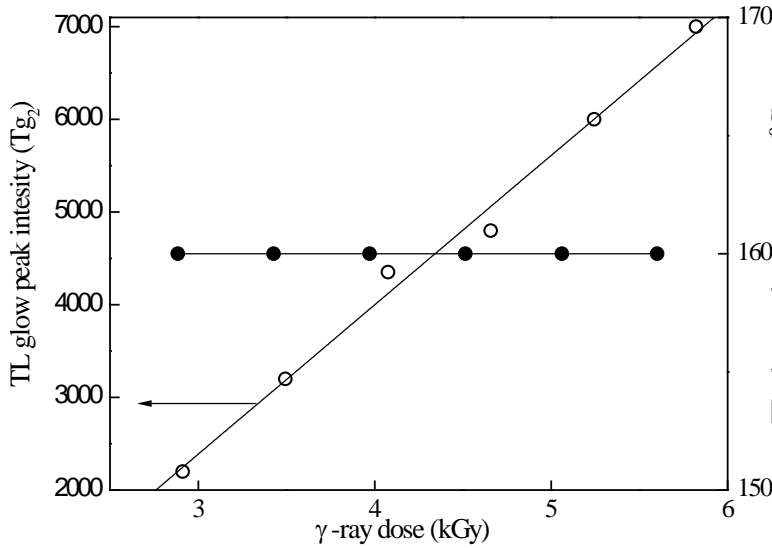


Figure 3. TL glow peak intensity and temperature as a function of dose

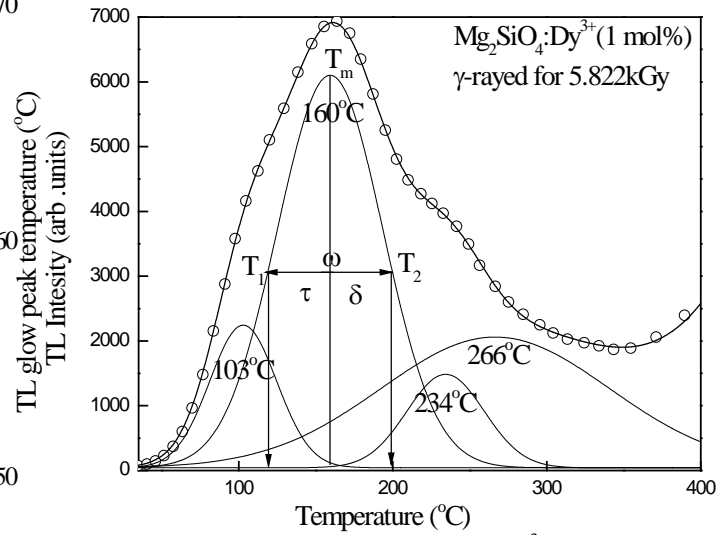


Figure 4. Deconvoluted TL glow curve in Dy<sup>3+</sup> doped Mg<sub>2</sub>SiO<sub>4</sub>

**Table 1.** Kinetic parameters obtained by using the glow curve shape method (modified by Chen) for a sample γ-rayed for 5.822 kGy .

T <sub>m</sub> (°C)	μ <sub>g</sub>	b	E <sub>τ</sub> (eV)	E <sub>δ</sub> (eV)	E <sub>ω</sub> (eV)	E <sub>a</sub> (eV)	n <sub>o</sub> (cm <sup>-3</sup> )	s (s <sup>-1</sup> )
103	0.491	2	0.683	0.723	0.706	0.704	6.42 x 10 <sup>3</sup>	9.28x 10 <sup>8</sup>
160	0.490	2	0.550	0.620	0.590	0.592	2.69 x 10 <sup>4</sup>	1.6 x 10 <sup>6</sup>
234	0.505	2	1.066	1.105	1.091	1.087	5.46 x 10 <sup>3</sup>	1.84 x 10 <sup>10</sup>
266	0.390	1	0.101	0.182	0.124	0.136	4.29 x 10 <sup>4</sup>	5.00 x 10 <sup>1</sup>