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Rare Earth Dy^{3+} Activated $MgSrAl_{10}O_{17}$ Phosphors for Luminescence

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ABSTRACT

$MgSrAl_{10}O_{17}:xDy^{3+}$ nanophosphors were fabricated by combustion method for different concentrations (x = 0, 0.0005, 0.001, 0.005, 0.01 and 0.02 mol) of Dysprosium (Dy). The synthesized nanophosphors were characterized by XRD, SEM, TEM, FTIR, PL and TL. The XRD (X-ray diffraction) showed crystalline hexagonal structure with preferred orientation of (107) plane. SEM (Scanning electron microscope) result shows the formation of nanosheets in irregular shape. TEM (transmission electron microscope) study revealed the nanoparticles within average diameter size of 30 nm. The FTIR (fourier transform infrared spectrum) shows absorption peaks in numerous regions. TL (thermo-luminescence) properties included TL glow curves and TL response for different concentrations of Dy after exposure of 700 Gy gamma rays. TL intensity was found to increase with increase in concentration of dopant Dy and was found to show best result for x=0.02. Further PL (photoluminescence) characterization of$MgSrAl_{10}O_{17}$:0.02 Dy^{3+} phosphor exhibits two main emission peaks at 484 and 575 nm due to Dy^{3+} ion, when excited with 350 nm wavelength.

Key words : SEM, Thermoluminescence, Nanophosphors, XRD, Kinetic Parameters.

1. INTRODUCTION

There is a rising demand for cost effective phosphors for utilization in display devices. A current technological improvement has offered different aluminates in luminescent applications as hosts for doping rare earth ions for best results. Rare earth activated aluminate based polycrystalline phosphors have many applications in electroluminescence panels, plasma display unit, light emitting diodes, fluorescent lamp, X-ray imaging plates, infra-red sensors , luminescence paints etc. In addition to these further applications of, long lasting afterglow phosphors are in radiation dosimetry, archaeological dating, and determination of natural radiation levels, environmental and retrospective dosimetry [1], [2]. The above stated applications are based on efficient radiation energy into the visible or near visible region of the electromagnetic spectrum. In contrast to their bulk counterparts, in nano- phosphor, a large number of charge carriers trapping centers have been produced at various energy depths. Due to high radiation resistance nano-phosphors play very significant role for the dosimetry of ionizing radiations. The Thermoluminescence technique is commonly used to monitor of ionizing radiations applied in medical, research and many other fields [3] .Green emitting phosphor $MgSrAl_{10}O_{17}$:Mn was successfully synthesized by combustion route in single phase and the luminescence, EPR (electron paramagnetic resonance) investigations have been published [4], [5]. Eu doped $MgSrAl_{10}O_{17}$ blue emitting phosphor was synthesized and characterized for study of luminescence properties [6]. Furthermore photoluminescence and energy transfer of Eu^{2+} and Cr^{3+} co doped $MgSrAl_{10}O_{17}$ have been explored [7] and defect centers in MgSrAl10O17: Sm has being studied [8]. Fabrication and characterization of green emitting Tb^{3+} doped $MgSrAl_{10}O_{17}$nanophosphor has also been evaluated [9]. Recently optical and EPR properties of Eu^{2+} and Mn^{2+} co-doped $MgSrAl_{10}O_{17}$ blue green light emitting phosphor have been reported [4]. Only a few researchers have published Dy doped $MgSrAl_{10}O_{17}$. Further the study of TL properties is another gap in the studies conducted earlier. In the present study, $MgSrAl_{10}O_{17}$: Dy phosphor was successfully synthesized by solution combustion method [10] and characterized for its applications as LED material and radiation dosimeters were evaluated.

2. EXPERIMENTAL DETAILS

2.1 Material and Methods

Dy doped $MgSrAl_{10}O_{17}$nanophosphors were fabricated by simple and low cost, combustion method. AR grade strontium nitrate ($Sr(NO_3)_2$), magnesium nitrate ($Mg(NO_3)_2$), aluminum nitrate ($Al(NO_3)_3$)and dysprosium oxide (Dy_2O_3) were used as starting materials. Urea was used as a fuel.

All the precursors were weighed as per the stoichiometric ratio and kept into a porcelain china dish. All the precursor materials along with its all gradients were mixed thoroughly with urea until it formed a white paste. The china dish containing this white paste was then kept in a muffle furnace, pre- heated at 550^{o}C.The combustion reaction takes place accompanied by a flame. The phosphor powder thus obtained was then crushed and annealed at 550^{o}C for 5 hours. After cooling at room temperature, the nanophosphors were collected and characterized for their structural, morphological and thermoluminescence (TL) properties. The reaction is as follows:-

$Mg(NO_3)6H_2O+Sr(NO_3)_2+10Al(NO_3)9H_2O+$
$85NH_2\text{-}CO\text{-}NH_2 \rightarrow MgSrAl_{10}O_{17} +96H_2O+85CO_2 +10\times2N_2$

2.2 Characterization

X-ray diffraction patterns were carried out for structural studies by using Rigaku X-ray diffractometer (Modal No. Mini Flex 600) with CuK_α source of radiation of x-ray wavelength (λ=1.54 Å). The XRD patterns were recorded in range of 10-85 degree in steps of 0.01 degree, in terms of intensity (I) versus 2-theta. The FTIR (Fourier transform infrared) spectrum was recorded by Perkin Elmer 105627 FTIR spectroscopy. SEM micrographs and TEM were used for surface morphological studies and images were obtained from scanning electron microscopy (model no. FE Quanta FEG 200) and TEM (transmission electron microscopy) (model no. JEOL 1230) respectively. The photoluminescence (PL) excitation and emission spectra of $MgSrAl_{10}O_{17}:xDy^{3+}$ (x = 0.02) were recorded in Shimadzu RF5309PC Spectro fluorophotometer. For TL studies, the samples were irradiated using Co^{60} gamma ray source to a dose 700 Gy and then their TL glow curves were recorded on Nucleonix TL1009I TL reader at heating rate of 5 K/sec.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

X-ray diffraction technique is used to analyze phase and crystallinity of grown material. Figure 1 shows XRD of host $MgSrAl_{10}O_{17}$ and Dy doped $MgSrAl_{10}O_{17}:Dy^{3+}$ samples. In sample A (Host $MgSrAl_{10}O_{17}$) diffraction peaks occurring at two theta values ~ 15.80°, 18.23°, 18.6°, 19.90°, 23.70°, 24.03°, 26.90°, 31.89°, 33.40°, 35.80°, 37.20°, 38.87°, 40.22°, 42.23°, 44.36°, 52.40°, 56.62°, 57.70°, 59.17°,62.07°,66.25° and 70.11° corresponding to the hexagonal structure of $MgSrAl_{10}O_{17}$ with space group P63/mmc (JCPDS 26-0879) [11]. Sample B (doped $MgSrAl_{10}O_{17}$ by Dy) shows same diffraction peaks which indicate that the addition of small amount of dysprosium (Dy) does not alter the crystal structure of $MgSrAl_{10}O_{17}$ (host lattice). This indicates that Dy^{3+} ion is successfully incorporated in the host lattice [12]. The lattice parameters of host lattice are calculated and approximated to be a = 0.516 nm, b = 0.895 nm and c = 0.992 nm. The value of unit cell volume is found to be V= 456.1 Å. By Scherrer's equation the average crystallite size of samples is calculated from full width half maximum (FWHM) to the corresponding diffraction peak (107) plane [13]-[15]. For the host lattice (sample A) and Dy doped lattice (sample B), it is found to be 35nm and 25 nm. The absence of extraneous peaks in the spectra indicates the formation of single phase $MgSrAl_{10}O_{17}$nanophosphers.

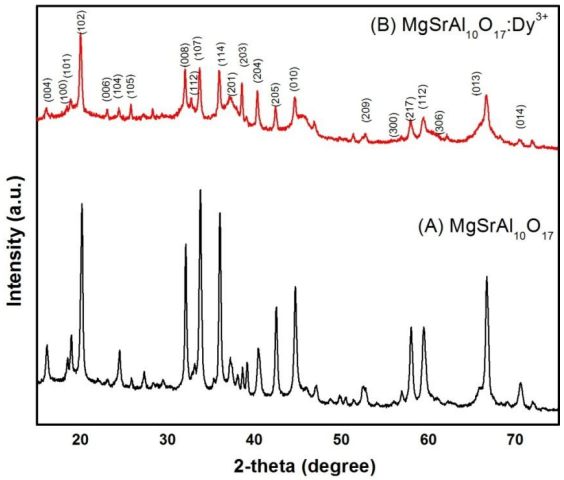

Figure 1: XRD patterns of host (A) and doped (B) $MgSrAl_{10}O_{17}$ samples

3.2 FTIR Analysis

The fourier transform infrared spectrum of dysprosium doped $MgSrAl_{10}O_{17}$phosphor is shown in Figure 2. Figure 2 consist of five peaks with energies ranging from 450 to 1200 cm^{-1} in addition different peaks at higher energies. The intensity of peak maximum at around 560, 667, 703, 775 and 1061 cm^{-1} corresponds to the formation of the $MgSrAl_{10}O_{17}$ phosphor and assigned to characteristic metal- oxygen vibrational mode in the hexagonal structure [16]. The maximum peak of metal-oxygen vibrational mode at 1061 cm^{-1} plays a important role in relaxation of Dy^{3+} ions. The low intensity peak around 3437 cm^{-1} could be referred to the stretching of O-H. The FTIR spectrum shows absorption in numerous regions: the

primary (a broad band) at about 3442 cm^{-1} which is due to O-H groups; the second one at about 1630 cm^{-1}, arising from the absorption of water (H_2O) vibrations and the peak at 1061 cm^{-1} originating due to the Al-O vibrations in the spinel block of β−alumina structure having symmetry A2u [17], [18].

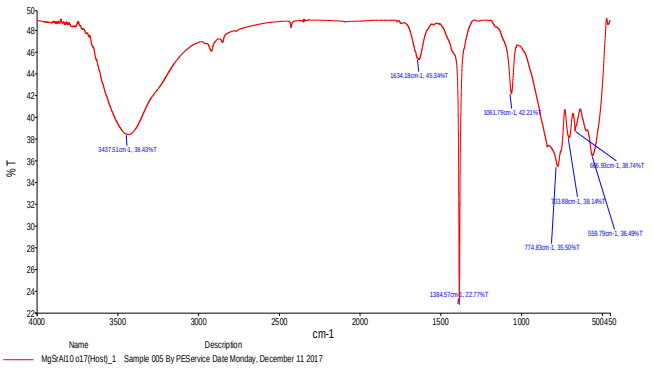

Figure 2: FTIR spectrum of host $MgSrAl_{10}O_{17}$ sample

3.3 SEM Results

Figure 3 shows low and high magnification SEM images of as prepared host and Dy doped $MgSrAl_{10}O_{17}$ nanophosphors.

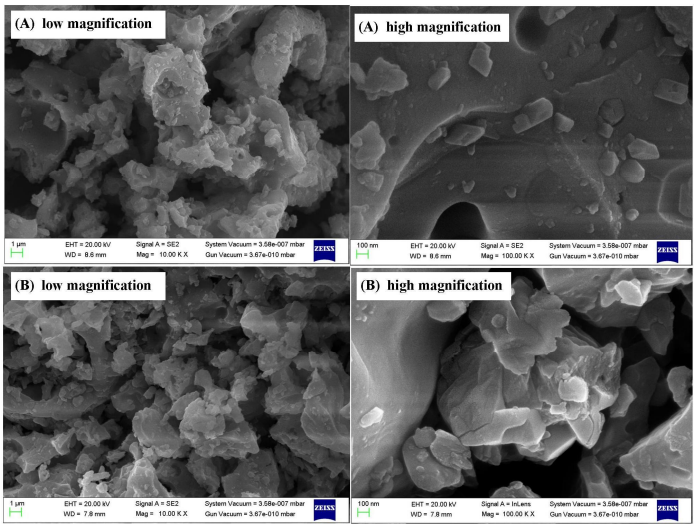

Figure 3: SEM images of samples (A) and (B) with low and high magnification.

The SEM micrographs depict non uniform and irregular shape and sizes of particles caused by non-uniform distribution of temperature and mass flow during process of combustion [13], [19]. At low magnification, the surface morphology of the samples (A) and (B) consist of foamy and porous agglomerated particles having sizes in range of 500 nm to 1 μm. It is clear from the images that the size of the particles increases as doped of Dy (dysprosium) in host material as shown in high magnification images (for samples A and B). The pores and voids are formed due to rapid release of gaseous by products during combustion process.

3.4 TEM & SAED Analysis

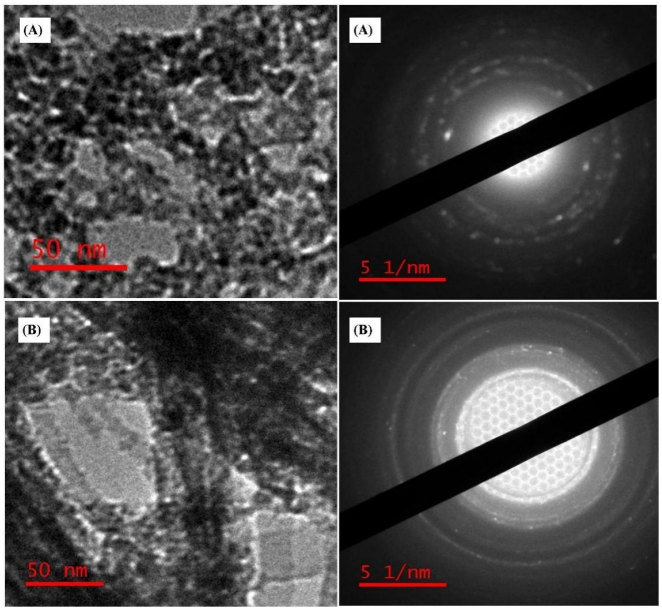

Figure 4: TEM images of samples (A) and (B) along with their SAED patterns.

Figure 4 shows the TEM images of host $MgSrAl_{10}O_{17}$ and doped $MgSrAl_{10}O_{17}:Dy^{3+}$ along with their SAED (selected -area electron diffraction) patterns. The TEM micrograph (for sample A) shows the presence of nano discs of size 20–30 nm whereas the particles size is increased having approximate diameter size of 30-40 nm (for sample B). The SAED patterns depict the better crystallinity for sample (A) than the doped sample (B). The results are in good agreements with the XRD and SEM results also [20].

3.5 TL Studies

TL glow curves of prepared $MgSrAl_{10}O_{17}:xDy^{3+}$ phosphor for different concentration (x = 0, 0.0005, 0.001,0.002, 0.01and0.02 mol) of Dy irradiated of 700Gy exposure of gamma rays were observed and are shown in Figure 5. It is clear that the $MgSrAl10O17:xDy^{3+}$ phosphor shows an isolated peak at around 180°C and also with increase in the activator concentration the intensity of TL peak keeps on increasing. It was observed that sensitivity of synthesized Dy activated $MgSrAl_{10}O_{17}$ was found to be 7 times greater than the pure host phosphor $MgSrAl_{10}O_{17}$. So, the dopant plays a significant role in describing the TL properties of phosphor. Dopant introduces dominantly deeper trapping levels in $MgSrAl_{10}O_{17}$ and as results the TL intensity is increased. The change in trap distributions may be because of lattice perturbation arises from incorporating of Dy in $MgSrAl_{10}O_{17}$ [21]

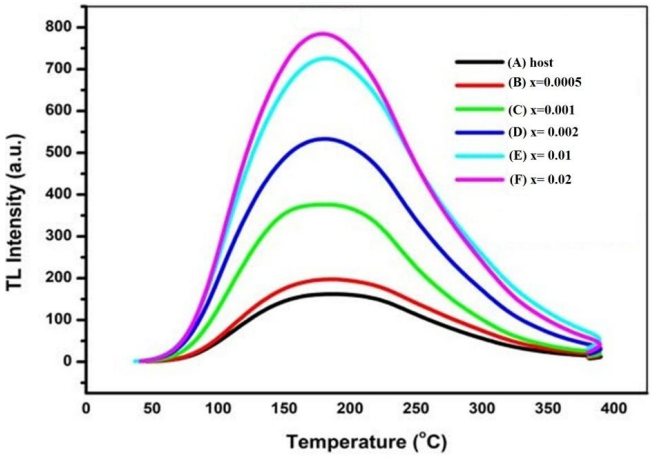

Figure 5: TL spectrum of host and doped $MgSrAl_{10}O_{17}$:xDy^{3+} at different concentration of x

An activator which serves as a luminescent center is surrounded by the non-luminescent host centers. As a consequence, the released charge carriers cannot recombine directly with the luminescent centers. Most likely the energy is transferred non-radiatively through the host lattice to the activator, gives characteristic emission after recombination. [21]. The determination of trapping parameters namely activation energy (E), order of kinetics (b) and frequency factor (s) have become more desirable to understand the properties and applications of the prepared phosphor [22], [23]. The parameters were calculated by using the peak shape method [1] which is based on the shape of glow curve.

3.5.1 Order of Kinetics

For the acquired glow curve, the order of kinetics (b) was determined by calculating the symmetry factor, as follows [1]:

$$\mu_g = \delta/\omega = (T_2 - T_m)/(T_2 - T_1) \qquad (1)$$

where, the total half intensity width also FWHM (full width at half maximum) is represented by $\omega = T_2 - T_1$. The high temperature half width is represented by $\delta = T_2 - T_m$. The symmetry factor of the glow curve having peak at 181^0C was found to be 0.51 by using (1) which suggests that the peak obeys second order kinetics.

3.5.2 Activation Energy

By using Chen's method [23], the activation energy (E) is calculated as

$$E = c_\gamma [kT_m^2/\gamma] - b_\gamma [2kT_m] \qquad (2)$$

where, the glow peak temperature is represented by Tm. The c_γ and b_γ are constants for second order kinetics having values of 1.71 and 0.0 respectively. γ denotes high temperature half width (δ) and k is the value of Boltzmann's constant. The activation energy for 181^0C glow peak of prepared sample, calculated was found to be 0.390 eV by using (2).

3.5.3 Frequency Factor

Frequency factor contains information on the probability to escape of electrons from their traps after exposure of ionizing radiation. It is closely related to the ability of phosphor to maintain the details of exposure information up to a certain time period and it is highly dependent on temperature. The frequency factor is calculated as [1], [23]:

$$\beta E[kT_m^2] = s[1+(b-1)2kT_m/E]\exp[-E/kT_m] \qquad (3)$$

where, β is the heating rate. The value of frequency factor(s) for 180^0C glow peak of grown sample is found to be 1.91×10^6 s^{-1}. It depends on different factors such as N_0, refers number of initial trap filling and its corresponding trap depth level associated to the irradiation, excitation dose, type and energy of the incident radiation.

3.6 PL studies

Figure 6 shows the PL excitation spectrum of Dy^{3+} activated $MgSrAl_{10}O_{17}$ phosphor, observed at the emission wavelength of 575 nm (4F9/2→6H13/2) as published in our previous report [24]. The PL excitation spectrum displays number of peaks at wavelength 324, 350, 387 and 454 nm are referred to the 6H15/2→4D7/2, (4M, 4I)15/2, 4I13/2 4G11/2 transitions, respectively resulting from f–f transitions of Dy3+ ion [25], [26].

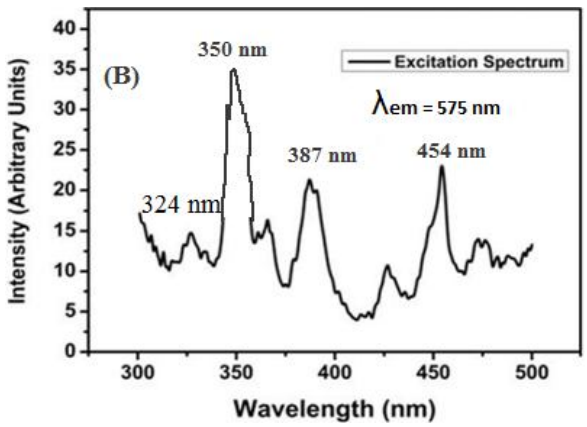

Figure 6: PL excitation spectrum of doped $MgSrAl_{10}O_{17}$sample (B)

At 350 nm excitation, the PL emission spectrum of $MgSrAl_{10}O_{17}$:0.02Dy^{3+} phosphor in the spectral region 400–650 nm is shown in Figure 7. This emission spectrum indicates two main emission peaks, at 484 and 575 nm Dy^{3+}

ion emission around 484 nm is ascribed to 4F9/2→6H15/2 is of allowed magnetic dipole origin [27], and the peak center at 575 nm is attributed to 4F9/2→6H13/2 transition due to electric dipole origin [28]-[30]. It was reported in the publication that emission of the Dy3+ ion luminescence is near to white region [31]. Consequently, $MgSrAl_{10}O_{17}$:0.02Dy^{3+} phosphor excited with 350 nm wavelength shows white emission which implies that Dy^{3+} activated $MgSrAl_{10}O_{17}$ phosphor may be a strong candidate in solid state lighting for white light emission.

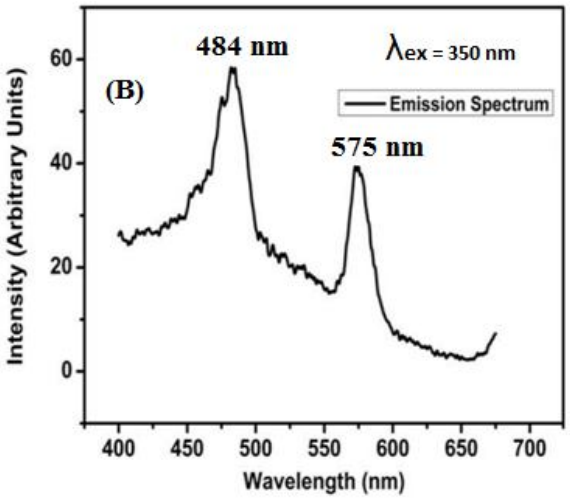

Figure 7: PL emission spectrum of doped $MgSrAl_{10}O_{17}$ sample (B)

4. CONCLUSION

$MgSrAl_{10}O_{17}$ doped with Dy was successfully prepared using combustion method for different concentrations of Dy (x = 0, 0.0005, 0.001, 0.002, 0.005, 0.01 and 0.02) and was found to show single peak TL glow curve at 180°C. From X-ray powder diffraction patterns the average particle size was found to be around 30 nm. The SEM micrograph shows the presence of micro to nano sized particles within the grains. The TEM micrograph shows the presence of nano discs of size 30-40 nm. The absorption bands in FTIR spectrum confirm the formation of desired phosphor material. In case of photoluminescence, MgSrAl10O17:0.02Dy3+ phosphor excited with 350 nm shows two main emission peaks, at 484 and 575 nm, attributed to 4F9/2→6H15/2 and 4F9/2→6H13/2 transition respectively. Thermo-luminescence behavior of prepared phosphor was studied after the gamma irradiation with Cobalt-60 source. The trapping parameters namely, order of kinetics (b) activation energy (E) and frequency factor (s) has been evaluated by PS (Peak Shape) method [32].

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