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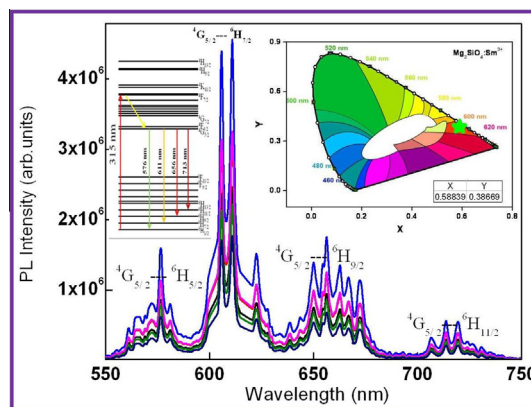
Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy

journal homepage: www.elsevier.com/locate/saaA single phase, red emissive $\text{Mg}_2\text{SiO}_4:\text{Sm}^{3+}$ nanophosphor prepared via rapid propellant combustion routeRamachandra Naik^{a,b}, S.C. Prashantha^{b,c,*}, H. Nagabhushana^{d,*}, S.C. Sharma^e, H.P. Nagaswarupa^c, K.S. Anantharaju^c, B.M. Nagabhushana^f, H.B. Premkumar^g, K.M. Girish^h^a Department of Physics, New Horizon College of Engineering, Bangalore 560103, India^b Research and Development Center, Bharathiar University, Coimbatore 641046, India^c Research Center, Department of Science, East West Institute of Technology, Bangalore 560091, India^d Prof. CNR Rao Center for Advanced Materials, Tumkur University, Tumkur 572103, India^e Chhattisgarh Swami Vivekananda Technical University, Bilai (CG) 493441, India^f Department of Chemistry, M.S. Ramaiah Institute of Technology, Bangalore 560054, India^g Department of Physics, Acharya Institute of Technology, Bangalore 560107, India^h Department of Physics, Dayanand Sagar Academy of Technology and Management, Bangalore 560082, India

HIGHLIGHTS

- Orthorhombic $\text{Mg}_2\text{SiO}_4:\text{Sm}^{3+}$ phosphor has been prepared using combustion technique at very low temperature (350 °C).
- Characteristic emission peaks of Sm^{3+} ion in the range 550–750 nm are recorded.
- The phosphor show different emissions due to Sm^{3+} corresponding to $^4\text{G}_{5/2} \rightarrow ^6\text{H}_j$ ($j = 5/2, 7/2, 9/2, 11/2$) transitions.
- Present phosphor was quite useful for WLEDs.

GRAPHICAL ABSTRACT

PL spectra, energy level diagram and CIE of $\text{Mg}_2\text{SiO}_4:\text{Sm}^{3+}$ nanophosphor.

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ABSTRACT

$\text{Mg}_2\text{SiO}_4:\text{Sm}^{3+}$ (1–11 mol%) nanoparticles were prepared by a rapid low temperature solution combustion route. The powder X-ray diffraction (PXRD) patterns exhibit orthorhombic structure with α -phase. The average crystallite size estimated using Scherer's method, W–H plot and strain-size plots were found to be in the range 25–50 nm and the same was confirmed by Transmission Electron Microscopy (TEM). Scanning electron microscopy (SEM) pictures show porous structure and crystallites were agglomerated. The effect of Sm^{3+} cations on luminescence of Mg_2SiO_4 was well studied. Interestingly the samples could be effectively excited with 315 nm and emitted light in the red region, which was suitable for the demands of high efficiency WLEDs. The emission spectra consists of four main peaks which can be assigned to the intra 4-f orbital transitions of Sm^{3+} ions $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{5/2}$ (576 nm), $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ (611 nm), $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{9/2}$ (656 nm) and $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{11/2}$ (713 nm). The optimal luminescence intensity was

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WLEDs
Solid state lighting

obtained for 5 mol% Sm^{3+} ions. The CIE (Commission International de l'Eclairage) chromaticity co-ordinates were calculated from emission spectra, the values (0.588, 0.386) were close to the NTSC (National Television Standard Committee) standard value of red emission. Coordinated color temperature (CCT) was found to be 1756 K. Therefore optimized $\text{Mg}_2\text{SiO}_4:\text{Sm}^{3+}$ (5 mol%) phosphor was quite useful for solid state lighting.

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Introduction

Nanomaterials and nanotechnology attracted several researchers working in the field of solid state lighting. Due to the nanometer regime, the physical properties were highly altered by the presence of significant number of surface atoms, quantum confinement, electronic states etc. which leads to the novel properties compared with their corresponding bulk phases [1]. Luminescence is one of the most important properties of rare-earth activated oxides and silicate nanoparticles [2–4]. Since the 4f electrons of trivalent rare-earth ions were well shielded from the surroundings, the emission transitions yield sharp lines in the spectra. Moreover, their 4f states only weakly interact with the host lattice, and therefore the energy differences was nearly constant, leading to almost the same light emission in different host lattices [5,6].

Rare earth [RE] ions were established much attention owing to their distinct electronic and optical characteristics arising from their 4f electrons. When rare earth ions, introduced into the suitable host, an efficient luminescent phosphors with high quantum yield, narrow bandwidth, large Stokes shifts, converting unusable UV to useful visible light, and ligand-dependent luminescence sensitization [7,8]. In particular, the rare earth doped luminescent materials attracted extensive attention due to the rising interests of the lighting industry and as result the materials were quite useful in solid state lighting (SSL) [9–11].

The phosphor materials used for SSL are the current research focus in the lighting industry which depends critically on the design of bright and stable phosphors. The application of WLED (white light emitting diode) as the next generation light source will change the life of human dramatically. The common WLED device was composed of chips that emit blue light and phosphors that can be excited by blue light to emit other colors. Since WLED devices were characterized by their high emission intensity, the quality of phosphors was vital to the performance of WLED device. WLEDs can save about 70% of the energy and do not need any harmful ingredient in comparison with the conventional light sources, such as incandescence light bulbs and the luminescent tubes. Therefore, WLEDs have a great potential to replace them and are considered as next generation solid state light devices [12,13].

Forsterite (Mg_2SiO_4); an important material in the magnesia-silica system and a member of the olivine family of crystals which has orthorhombic crystalline structure in which Mg^{2+} occupies two non equivalent octahedral sites: one (M1) with inversion symmetry (CI) and the other (M2) with mirror symmetry (CS). The material has some essential properties, such as high melting point, chemical stability even at high temperatures, vast electrical and refractory characteristics, as well as good mechanical properties, bioactivity and biocompatibility [14]. Therefore, it has found commercial applications in many industrial areas, e.g. electronics as insulators working at high frequencies [15], refractory industry [16], advanced technologies such as SOFC (solid oxide fuel cells) [17], biomedicine [18] and luminescent technology [19].

Efficiency of a nanophosphor depends on a host material and also on the improved synthetic routes. Pure and doped Mg_2SiO_4 nanoparticles were synthesized by different chemical routes. The preparation of forsterite via solid-state reactions usually requires

high temperature and long reaction time while the solution combustion process with the proper selection of fuel, was a rapid and precise process to achieve nano size compound and also reduces crystallization temperature and prevent from phase segregation during heating because it can not only be implemented in several minutes at a relatively low temperature but also allows molecular-level of mixing, high degree of homogeneity and uniform doping of trace amounts of rare earth ions in a single step [20].

The nanoparticles of rare earth ions doped Mg_2SiO_4 shows excellent photo, thermo and ionoluminescence properties. Literature reveals that the Mg_2SiO_4 doped with various rare earth ions find wide range of applications [1,14,19–24]. Table 1 gives review of literature on Mg_2SiO_4 host doped with various rare earth ions.

Herein, we report a facile solution combustion synthesis, structural characterization and photoluminescence properties of $\text{Mg}_2\text{SiO}_4:\text{Sm}^{3+}$ (1–11 mol%) nanoparticles. The advantages of the present work are based on the following three aspects. (i) A potential luminescence host because of obvious energy transfer from host to Sm^{3+} ions. The fact that Mg^{2+} ions have no 4f electron implies that Mg_2SiO_4 can be a potential luminescence host material due to strong light absorption through charge transfer (CT) from O–Mg. If the energy transfer was achieved from the CT state of Mg_2SiO_4 to the doped rare earth ions, the characteristic emissions of rare earth ions were expected to be observed. (ii) Optimized luminescence, recently, we investigated photoluminescence properties of Eu^{3+} (1–11 mol%) activated Mg_2SiO_4 [20] in which case maximum intensity observed for 11 mol% Eu^{3+} . However, in the present work, luminescence intensity saturated at 5 mol% Sm^{3+} in the same host material. (iii) Potential application – then as prepared $\text{Mg}_2\text{SiO}_4:\text{Sm}^{3+}$ nano scale samples with strong luminescence may be suitable for use in solid state lighting. It was known that one of the challenges for UV-LEDs was to develop stable, efficient and environmentally friendly low temperature synthesized and low energy consumed nanophosphors that can be excited between 300 and 400 nm [25–26] which was expected to meet the demands of solid state lighting.

Experimental

Synthesis

The raw materials namely magnesium nitrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$: 99.99%, Sigma–Aldrich), fumed silica (SiO_2 , Sigma–Aldrich) and samarium nitrate ($\text{Sm}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$: 99.99%, Himedia) were the sources of Mg, Si and Sm, respectively. Laboratory prepared oxalyl dihydrazide (ODH: $\text{C}_2\text{H}_6\text{N}_4\text{O}_2$) was used as the fuel. Stoichiometric amounts of the above combination were dissolved in a minimum quantity of doubled distilled water in a cylindrical Pyrex dish and mixed thoroughly using magnetic stirrer for about 5 min. The stoichiometry of the redox mixture used for the combustion synthesis was calculated based on the total oxidizing and reducing valencies of the compounds [27]. The Pyrex dish containing this solution was placed in a pre-heated muffle furnace maintained at 350 ± 10 °C. After a while the mixture underwent decomposition, which results in the liberation of large amounts of gases (usually CO_2 , H_2O and N_2). This was followed by a spontaneous ignition

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