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Synthesis and optical characterization of novel $Sr_3Ga_2O_6:Eu^{3+}$ phosphor

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Abstract

Alkaline earth metal gallets have been identified as an important ceramic material. The crystal chemistry of many of these gallets is well explored; however, very rare studies regarding optical properties of rare earth (RE) ions doped in such gallets, particularly in $Sr_3Ga_2O_6$ host, have been carried out. The present study reports on synthesis and characterization of novel $Sr_3Ga_2O_6:Eu^{3+}$ phosphors. The phosphors have been synthesized using a conventional solid state reaction method. Crystal structure, morphology and luminescence properties (excitation, emission and CIE coordinate) of these phosphors have been studied as a function of sintering temperature and Eu^{3+} concentration. X-ray diffraction study reveals that the phosphor sintered at low temperature (900 °C) contains an impurity phase which is removed at higher sintering temperatures and results into cubic crystalline phase of $Sr_3Ga_2O_6$. Particle size of the phosphor increases with an increase in sintering temperature which results to a red shift in the peak position of excitation band lying in a broad range from 250 to 370 nm. Optimum emission intensity is attained for 0.12 mol% concentration of Eu^{3+} ions; above this concentration, a quenching in emission intensity is observed.

Keywords: $Sr_3Ga_2O_6$; Photoluminescence; Red emitting phosphor

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1. Introduction

Illumination technology has become indispensable in our daily life and thus considerable efforts are being made to develop suitable luminescent materials for lighting and display applications [1–3]. However, still there is a quest for suitable exotic photonic materials with better performance and low cost. Recently, considerable attention has been paid on rare earth (RE) doped inorganic materials (also called phosphors), due to the characteristic optical features of RE ions (viz. narrow emission band widths (<10 nm), long luminescence lifetime (μs–ms range), better quantum yield, etc) in such hosts. These inimitable optical features make them highly suitable for lighting and display devices [4–6].

In the midst of different RE doped phosphors, materials doped with trivalent Eu ions have been found much interesting due to simple lower energy level scheme of europium. Moreover, Eu^{3+} ions are well known for their intense, narrow and monochromatic red emission as a result of $^5D_0 \rightarrow {}^7F_2$ transition. Thus, phosphors based on Eu^{3+} doped RE sesquioxides, such as yttrium oxide and gadolinium oxide, have been widely studied for red light emission usable for modern optoelectronic devices [7–9]. However, in spite of several merits, these sesquioxides based phosphors are not an ideal UV (ultra-violet) sensitizer to activate certain RE^{3+} ions (viz. Eu^{3+}) for their emission in visible region. Hence, it is imperative to search and develop new families of red-emitting phosphors with efficient absorption in the UV or blue region.

Different hosts with efficient absorption in the UV or blue region have been already tried and reported.

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Choi et al. [10] reported the broad band excitation (200–350 nm) in YVO_4 matrix and used it to activate Eu^{3+} ions for red emission. Omkaram and Buddhudu [11] used the broad charge transfer band (CTB) excitation of $MgAl_2O_4$ in 210–300 nm region to activate Dy^{3+} ions for yellow emission. Similarly, gallium oxide (Ga_2O_3) has also gained much attention as a new phosphor host material due to its strong absorption in UV region. In addition to this, ceramics such as gallets (double oxides based on alkaline earth metal (viz. Sr or Ca) oxides and gallium oxides) have been identified as an excellent and cost effective ceramic material for different applications [13,14].

Crystal chemistry of many of these alkaline earth metal gallets is well explored. According to the phase equilibrium studies of Kobzareva et al. [15] seven different strontium gallets are possible. However, very rare studies regarding optical properties of RE ions doped in such gallets, particularly in $Sr_3Ga_2O_6$ host, have been carried out [16]. In this paper, authors have investigated a new and thermally stable $Sr_3Ga_2O_6$:Eu^{3+} phosphor. The main aim is to search and develop a new host, which itself has its strong absorption in UV region and can act as a sensitizer to activate Eu^{3+} ion for red emission. This type of new phosphor could be useful for lighting and display applications. The phosphors have been synthesized using a conventional solid sate reaction method and characterized for their structure and optical properties. Investigated phosphors show a broad excitation band lying in the UV region (250–370 nm) and emit excellent red light with high color purity.

2. Experimental

2.1. Synthesis of $Sr_3Ga_2O_6$:Eu^{3+} phosphor

Powder samples of $Sr_3Ga_2O_6$:Eu^{3+} phosphors were synthesized by conventional solid state reaction method. Doping level of Eu^{3+} was kept 0, 0.04, 0.08, 0.12 and 0.16 mol%. High purity chemicals strontium carbonate ($SrCO_3$, ALDRICH, 99.9%), gallium oxide (Ga_2O_3, ALDRICH, 99.99%) and europium oxide (Eu_2O_3, ALDRICH, 99.9%) were used as starting materials. These chemicals were taken in stoichiometric amounts and then they were finely ground in an agate mortar with a pestle on adding a little amount of acetone. The mixture was first taken in an alumina crucible and calcined at 500 °C for 4 h. Then after, the calcined powder was sintered at different temperatures (900 °C, 1000 °C, 1100 °C and 1200 °C) for 4 h in air atmosphere in an electric tubular furnace. Sintered materials were allowed to cool down to room temperature. After these procedures, white color powder samples of the $Sr_3Ga_2O_6$:Eu^{3+} phosphors were obtained and used for further study.

2.2. Characterization

The crystal purity of the prepared powder samples was examined by X-ray diffration (XRD) analysis using a high-resolution XRD system (SHIMADZU, XRD-6000) with CuKα1 radiation ($\lambda = 1.540562$ Å). X-ray photoelectron (XPS) spectroscopic measurements were carried out with an ESCALAB 250 XPS spectrometer, using MgKα as the X-ray source. Surface morphologies of the samples were investigated by a field emission gun scanning electron microscope (FESEM, Quanta 200 FEG) operated at 15 kV. Photoluminescence excitation (PLE) and emission (PL) measurements were carried out using spectrophotometer (model FS-2) equipped with a monochoromator and 150 W continuous Xenon lamp.

3. Results and discussion

3.1. X-ray diffraction (XRD) analysis

The XRD patterns of the phosphor samples prepared at different sintering temperatures are shown in Fig. 1. The sample sintered at 900 °C shows the presence of an impurity phase. The impurity phase is assigned as $SrCO_3$ (JCPDS file no. 71-2393) and corresponding diffraction peaks are marked with an astrix. Remaining diffraction peaks in the XRD pattern could be assigned well due to the cubic crystalline phase of $Sr_3Ga_2O_6$ (JCPDS file no. 24-1200). Sintering above 900 °C removes the impurity phase due to $SrCO_3$. However, the intensity of diffraction peaks decreases for the sample sintered at 1100 °C and 1200 °C. This indicates a decrease in crystallinity of the phosphor samples sintered at 1100 °C and 1200 °C. The decrease in crystallinity can also be verified through the FWHM (full width at half maximum) of the diffraction peaks.

In the present case, it is expected that some of the gallium ions are evaporated at high sintering temperature which may cause a decrease in the crystallinity. The evaporation of Ga ions is also supported by a decrease in the intensity of XPS peak corresponding to Ga ($^2P_3$) at 1117.7 eV and 1114.8 eV, shown in Fig. 2. It is already reported that

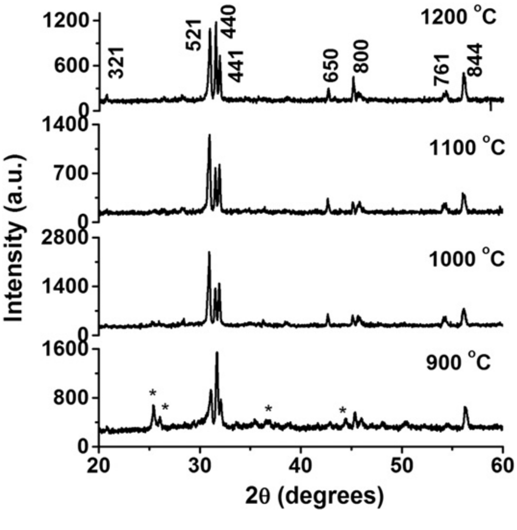

Fig. 1. X-ray diffraction (XRD) patterns of $Sr_3Ga_2O_6$ phosphor synthesized at various sintering temperatures.

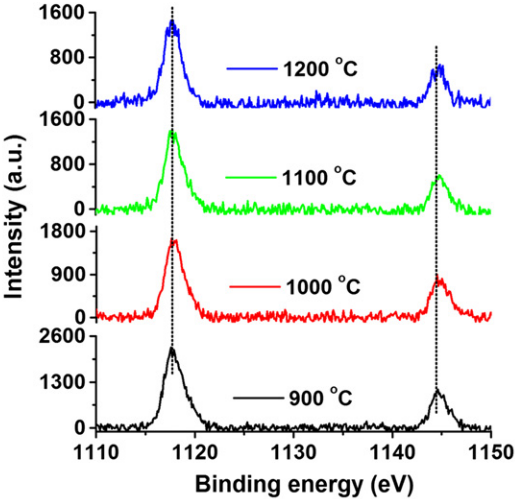

Fig. 2. X-ray photoelectron spectroscopy (XPS) spectra of $Sr_3Ga_2O_6$ phosphor synthesized at different sintering temperatures showing a variation in the intensity of Ga, $^2P_3$ peak.

Ga_6O_{18} tetrahedral rings are the main building unit in the crystal structure of $Sr_3Ga_2O_6$. These tetrahedral rings within the structure remain connected by Sr ions. The rings in $Sr_3Ga_2O_6$ structure remain extremely puckered, such that the Ga atoms at the center of the six tetrahedra are located near the corner of a cube [17]. As the sintering temperature is increased, some gallium ions from the tetrahedral rings are evaporated (as is supported by XPS analysis also) which may result in a significant decrease in the crystallinity of the phosphor. The effect of evaporation of Ga ions on the overall crystal structure would be an interesting problem for crystallography and needs a detailed Rietveld refinement of XRD data, but beyond the scope of the present study.

3.2. *Microstructure analysis*

FESEM has been used to study the morphology and particle size of the phosphor powder both for the samples synthesized at different sintering temperatures as well as for the samples prepared with various Eu^{3+} concentrations. Fig. 3 shows the FESEM images of $Sr_3Ga_2O_6:Eu^{3+}$ phosphor (Eu^{3+}: 0.12 mol%) synthesized at 900 °C, 1000 °C, 1100 °C and 1200 °C sintering temperatures. The particles show polyhedral shape morphology with agglomerated nature. The particle size for almost all the phosphor powders shows a large variation from sub-micrometers to a few micrometers. A significant increase in particle size is observed as the sintering temperature increases. The particle size varies from 0.5 to 1 μm for the sample sintered at 900 °C, 1 to 2 μm for the sample sintered at 1000 °C, 1 to 3 μm for the sample sintered at 1100 °C, and from 3 to 5 μm for the sample sintered at 1200 °C. However, no such variation in particle size is observed if the concentration of Eu^{3+} ions is varied. Fig. 4 shows the microstructures of the samples with different concentrations of Eu^{3+} (sintered at 1000 °C). No significant variation either in the particle size or in the surface morphology was observed.

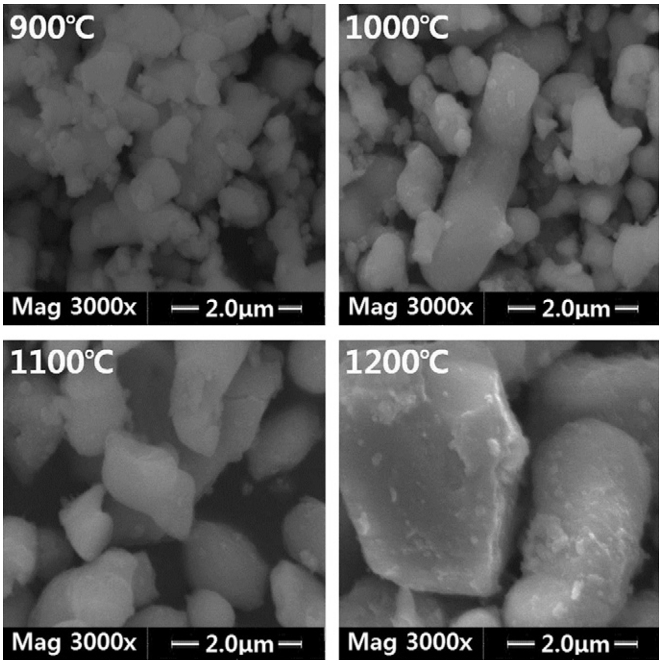

Fig. 3. Field emission scanning electron microscopy (FESEM) images of $Sr_3Ga_2O_6:Eu^{3+}$ phosphor (Eu^{3+}: 0.12 mol%) sintered at (a) 900 °C, (b) 1000 °C, (c) 1100 °C and (d) 1200 °C.

3.3. *Photoluminescence characteristics*

Luminescence characteristics were analyzed by photoluminescence excitation (PLE) and emission (PL) measurements as a function of sintering temperature and Eu^{3+} concentration.

3.3.1. *Effect of sintering temperature*

Fig. 5 shows PLE and PL spectra of $Sr_3Ga_2O_6:Eu^{3+}$ phosphor (Eu^{3+}: 0.12 mol%) synthesized at various sintering temperature. The PLE spectra ($\lambda_{em}=618$ nm) for the sample synthesized at 900 °C, shows a broad band structure from 250 to 375 nm followed by a few relatively weak and sharp bands. The peak position of the broad band appears around 300 nm. The band with its maximum around 300 nm could be attributed due to the Eu^{3+} to O^{2-} charge-transfer band (CTB) lying in the band gap region of the host matrix. The other additional broad excitation band with its peak position at 250 nm (towards the higher energy side of CTB band) could be assigned due to electron transfer transition i.e. excitonic band. This band becomes prominent at higher sintering temperature (1200 °C). The presence of electrons and holes in the p state of oxygen lying in close proximity with Sr^{2+} site may result such bands. Dhanaraj et al. [18] have also reported the presence of both the excitonic and the charge transfer band in Eu^{3+} doped Y_2O_3 phosphor and have concluded that the CTB absorption process is a dominating process. Divalent Eu could also give such a broad band feature in PLE spectrum but the absence of any characteristic emission feature in PL spectrum (recorded in 400–700 nm, not shown here) rules out this possibility.

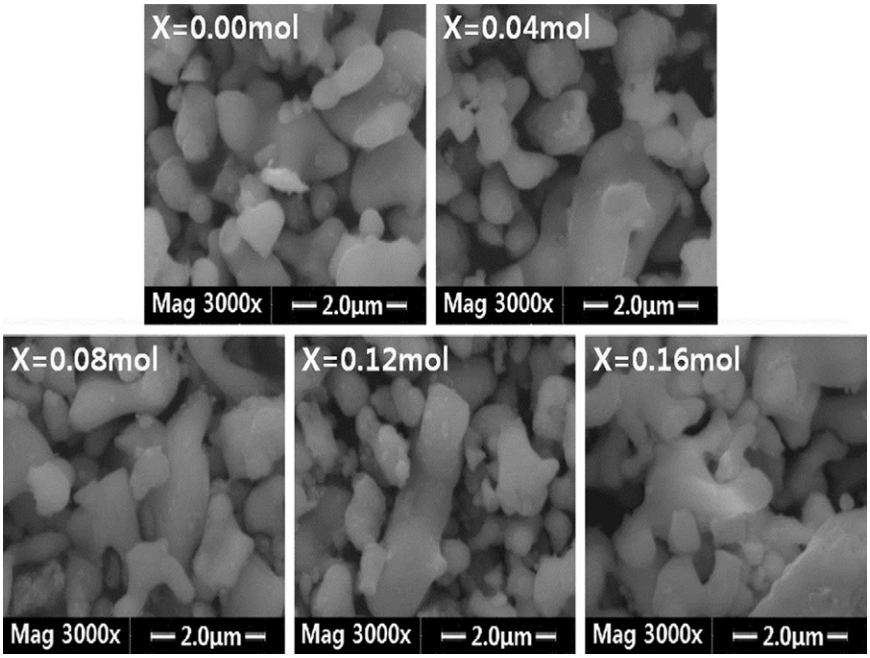

Fig. 4. Field emission scanning electron microscopy (FESEM) images of $Sr_3Ga_2O_6:Eu^{3+}$ phosphor (sintered at 1000 °C) with various concentration of Eu^{3+}.

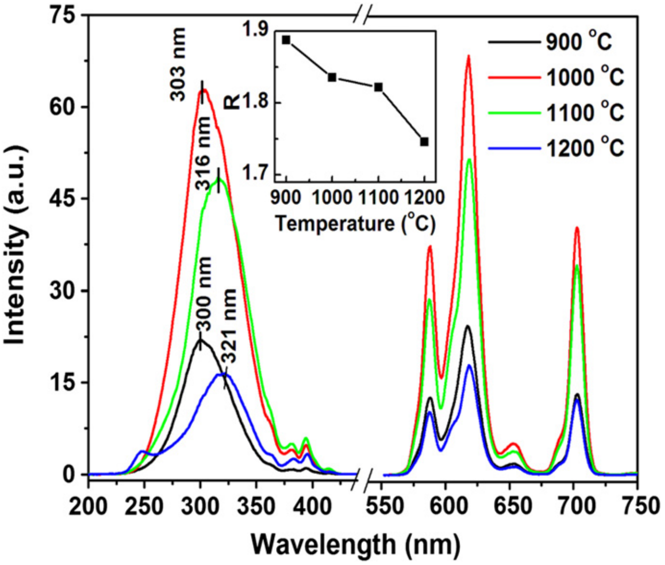

Fig. 5. Comparison of photoluminescence excitation (PLE) and emission (PL) spectra for $Sr_3Ga_2O_6:Eu^{3+}$ phosphor (Eu^{3+}: 0.12 mol%) synthesized at various sintering temperatures. Inset shows the intensity ratio (R, for $^5D_0\rightarrow{}^7F_2$ to $^5D_0\rightarrow{}^7F_1$ transition) versus sintering temperature graph.

Other sharp weak bands could be assigned due to the well known f–f (4f) transitions, from ground state ($^7F_0$) to various excited states of Eu^{3+} ions.

As the sintering temperature is increased two significant differences are marked in PLE spectra. The first change is observed in the intensity of the PLE bands, which shows an increase for the sample sintered at 1000 °C. However, subsequently, the PLE intensity started to decrease for the samples sintered above 1000 °C. The decrease in intensity of the excitation peak could be related to a decrease in the crystallinity of the phosphor powder. It has already been noted in the XRD analysis that the intensity of the diffraction peaks decreases for the samples sintered at 1100 °C and 1200 °C compared to samples sintered at 1000 °C. The second change is observed in the peak position of the broad PLE band. It shows a red shift (a shift towards longer wavelength side) with an increase in sintering temperature. A red shift of almost 21 nm is noted for the sample sintered at 1200 °C. This could be attributed to an increase in particle size with sintering temperature [18].

The PL spectrum ($\lambda_{exc}=300$ nm) of $Sr_3Ga_2O_6:Eu^{3+}$ phosphor (Eu^{3+}: 0.12 mol%) shows four characteristic emission bands centered at around 587 nm, 618 nm, 654 nm and 703 nm which could be assigned to arise due to $^5D_0\rightarrow{}^7F_1$, $^5D_0\rightarrow{}^7F_2$, $^5D_0\rightarrow{}^7F_3$ and $^5D_0\rightarrow{}^7F_4$ transitions of Eu^{3+} ion, respectively. The strongest emission peak located at around 618 nm corresponds to the forced electric dipole $^5D_0\rightarrow{}^7F_2$ transition of Eu^{3+} ion. The intensity of the PL bands follows a similar trend, as that of PLE, with a variation in the sintering temperature. The maximum emission is achieved for the sample sintered at 1000 °C, above this temperature a decrease in the emission intensity is noted. However, no change, either in peak position or in the shape of transition bands, is marked.

Transition $^5D_0\rightarrow{}^7F_1$ is used as a reference to judge the environmental asymmetry of the rare earth because it is allowed by the magnetic dipole transition and its intensity is independent of the environment whereas the hypersensitive transition ($^5D_0\rightarrow{}^7F_2$) strongly depends on it. The intensity ratio (R) of $^5D_0\rightarrow{}^7F_2$ to $^5D_0\rightarrow{}^7F_1$ transition (also called monochromaticity) is commonly used as a measure of the

RE site symmetry [19]. Lower value of R signifies higher symmetry and vice versa. The inset in Fig. 5 shows a decrease in the value of R as the sintering temperature is increased, which signifies that the symmetry around the Eu^{3+} ion increases with an increase in sintering temperature. This indicates that Eu^{3+} ions are situated on the Sr^{2+} site.

Replacement of Sr^{2+} ions by Eu^{3+} ions in $Sr_3Ga_2O_6$ matrix is also supported by the Shannon effective ionic radii of cations [20] effect, which states that a substituting ion should be of almost similar radii to that of host ion. So, it is clear that the Eu^{3+} (ionic radius 1.066 Å) would like to substitute the Sr^{2+} ions among the two possible sites i.e. Sr^{2+} (ionic radius 1.18 Å) and Ga^{3+} (ionic radius 0.62 Å) because of their similar ionic radii. To keep the electroneutrality of the compound, two Eu^{3+} ions would substitute for three Sr^{2+} ions. As a result, two positive defects of $[Eu_{Sr}]^*$ and one negative Sr^{2+} vacancy $[V_{Sr}]''$ would be created by each substitution for every two Eu^{3+} ions in the compound. By thermal stimulation, electrons of the $[V_{Sr}]''$ vacancies may reduce Eu^{3+} to Eu^{2+}, but even if this possibility exist, the oxygen atom in the structure which is in direct contact with Sr ion would oxidize these divalent ions to trivalent europium ions.

3.3.2. *Effect of Eu^{3+} concentration*

In the effect of sintering temperature, we observed that the peak of excitation maximum shows a red shift with an increase in sintering temperature. So in order to study the effect of Eu^{3+} concentration authors selected two samples, first sample sintered at 1000 °C (with its excitation maximum around 303 nm, see Fig. 5) and the other sintered at 1200 °C (with its excitation maximum around 321 nm, see Fig. 5). Fig. 6 shows PLE and PL spectra of $Sr_3Ga_2O_6$:Eu^{3+} phosphor sintered at (a) 1000 °C and (b) 1200 °C with a variation in Eu^{3+} concentration. The shape of PLE spectra for the phosphor sintered at 1000 °C remains unaltered with a variation in concentration of Eu^{3+}. However, for the case of phosphors sintered at 1200 °C, the shape of PLE spectra shows a significant change, mainly with the peak located at around 250 nm, with an increase in Eu^{3+} concentration. The change in the peak position of the PLE band located at 250 nm is obviously due to occupancy of Sr^{2+} sites by Eu^{3+} ions.

On the other hand, the intensity of the PLE peaks changes significantly when Eu^{3+} concentration is increased and as a result a variation in the PL intensity is also observed. The dependence of PL emission intensity on Eu^{3+} concentration is shown in insets of Fig. 6. Insets in Fig. 6(a) and (b) both depict that the emission intensities increase with Eu^{3+} content until a maximum intensity is reached for 0.12 mol% concentration, and then they decrease due to concentration quenching caused by the well known resonant energy transfer among Eu^{3+} ions [21].

The color purity of the material can be realized in terms of mathematically driven coordinates, called Commission International De I'Eclairage (CIE) chromaticity coordinates. Fig. 7 shows the CIE chromaticity diagram for

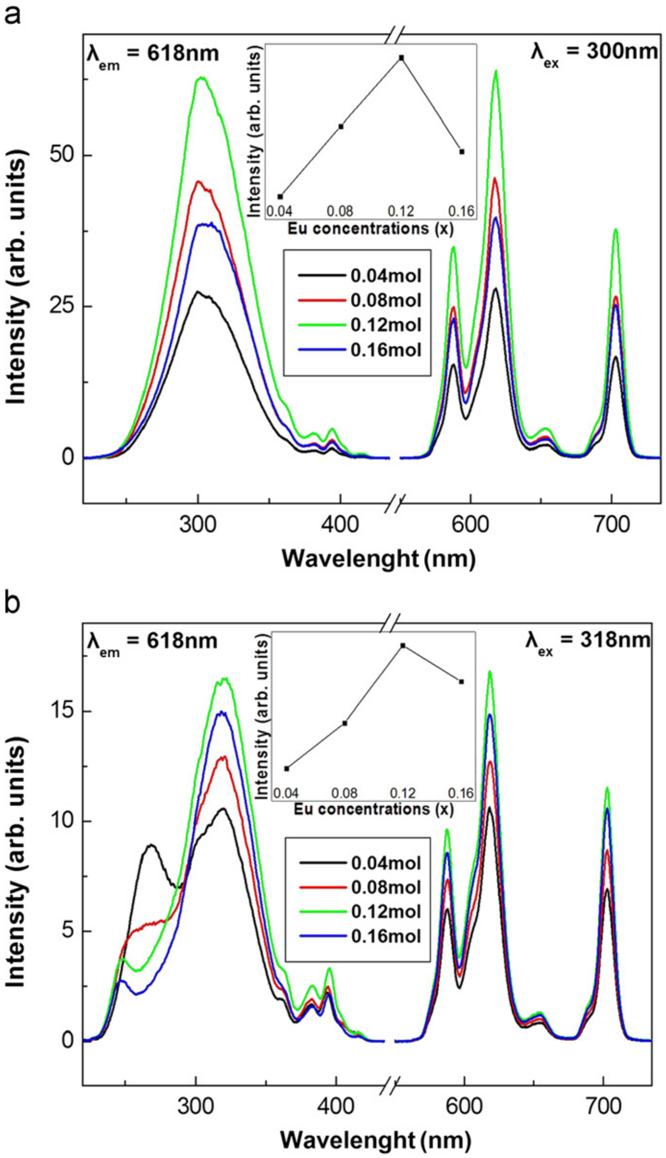

Fig. 6. Photoluminescence excitation (PLE) and emission (PL) spectra of $Sr_3Ga_2O_6$:Eu^{3+} phosphor with various Eu^{3+} concentrations sintered at (a) 1000 °C and (b) 1200 °C. Inset to figure (a) and (b) both shows the concentration quenching effect.

$Sr_3Ga_2O_6$:Eu^{3+} phosphor (Eu^{3+}: 0.12 mol%) sintered at different temperatures, under UV (300 nm) excitation. The color co-ordinates for samples sintered at 900 °C and 1000 °C almost coincides (0.65, 0.33) while for 1100 °C and 1200 °C they show a little variation and are found to be (0.66, 0.31) and (0.67, 0.30), respectively. All the CIE coordinates lie well within the red region, and suggest that the investigated phosphor could serve as a red emitting phosphor for display/lamp applications.

4. Conclusion

Various concentration of Eu^{3+} ion-doped $Sr_3Ga_2O_6$ phosphors have been synthesized at different sintering temperatures and characterized for structure and optical properties. The best crystallinity is achieved for the sample sintered at

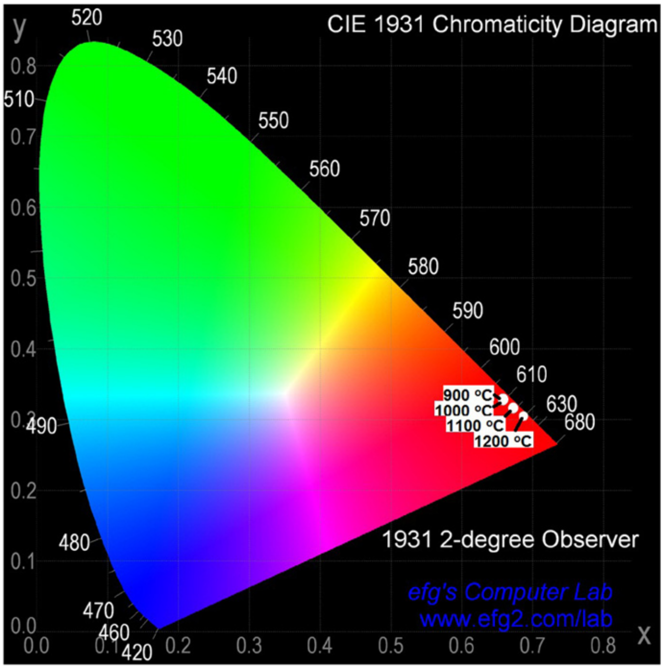

Fig. 7. CIE chromaticity diagram for $Sr_3Ga_2O_6$:Eu^{3+} phosphor (Eu^{3+}: 0.12 mol%) under UV (300 nm) excitation for different sintering temperatures.

1000 °C, with a particle size on the order of 0.5–5 μm. The particle size increases with an increase in sintering temperature, which results in a red shift of the peak position of excitation maxima (monitored for $^5D_0 \rightarrow {}^7F_2$ transition of Eu^{3+}). The sample sintered at 1000 °C gives the highest emission intensity for 0.12 mol% concentration of Eu^{3+} ions, above this concentration, a quenching in emission intensity is observed. CIE coordinates for the phosphors lie well within the red region which is significant for the development of a color pure phosphor.

Acknowledgments

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