Effects of Synthesis Methods on the Characteristics of Material Sr₃Y_{1.95}Ge₃O₁₂: 0.05Eu³⁺ (SYGO: Eu³⁺) for White LED Phosphors Applications

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Abstract

Solid-state reaction, co-precipitation, and sol-gel procedures were used to synthesize the $Sr_3Y_{1.95}Ge_3O_{12}:0.05Eu^{3+}$ (SYGO: Eu^{3+}) garnet structure. The morphological structure and photoluminescence properties of the samples were studied. Phosphors have morphologies and particle sizes that vary depending on the preparation. According to the X-ray diffraction pattern measurement, the sol-gel sample only appeared to exhibit the SYGO: Eu^{3+} characteristic that it shows the cubic structure belonging to the la3d (230) space group. In comparison to the two approaches of co-precipitation and solid-phase reaction, the sol-gel method has a more single-phase structure. Under the excitation wavelength of 395 nm, all produced samples emitted red light, with the greatest emission peak at 612 nm, corresponding to the ⁵D₀—⁷F₂ transition of Eu^{3+} ions in the SYGO host lattice. The color coordinates of the sample SYGO: Eu^{3+} fabricated by the sol-gel method are x = 0.65 and y = 0.35. Therefore, the SYGO: Eu^{3+} based on sol-gel method's synthesis is a promising phosphor for the application of White LED.

Keywords: $Sr_3Y_{1.95}Ge_3O_{12}$: 0.05Eu³⁺ phosphors, SYGO:Eu³⁺, hydrothermal, co-precipitation, solid-state reaction.

1. Introduction

When compared to conventional light sources, phosphor-converted LED (pc-LED) is regarded as the next generation of solid-state light sources because of its energy savings, good durability, high efficiency, and environmental friendliness [1]. A phosphor layer is coated on top of the LED chip to create a pc-LED (Ultraviolet chip or blue chip). There have been numerous methods for producing white LEDs until now (White LEDs). The first method is to cover nearultraviolet (NUV) chips with light-emitting phosphors in the red, green, and blue spectral areas [2]. Combining a blue LED chip (the blue chip) with a phosphor that generates light in the green and red spectral areas is the second method for producing white light. Alternatively, blue chip could be combined with phosphor material that emits green, yellow, and red [3]. However, because UV chips are relatively expensive, scientists have been concentrating their efforts in recent years on studying and manufacturing phosphors for blue chips.

White LEDs on the market today are primarily coated with phosphor YAG: Eu^{3+} on blue chips. Depending on the thickness of the phosphor coating, the color of the White LED will change from warm white to cold white.

Due to the lack of a color rendering index (CRI>80) and a high color temperature (CCT>4500 k), this method of White LED production has some disadvantages of red light in the spectrum (> 600 nm). There have been many publications that have shown that a red line emitter emitting at 610–615 nm has good luminous efficacy and color rendering. Therefore, it is necessary to study the synthesis of red phosphor in order to apply White LED fabrication [4].

The host lattice belongs to the rare-earth iondoped garnet structure, which has been shown to be a material with good luminescence properties, suitable as a phosphor for White LED fabrication, due to its structural, physical, and chemical properties. and their thermal stability [5]. The garnet structure has the general formula $A_3B_2X_3O_{12}$ where A = Sr, Ca, B = Y, Sc, Al, Ga, In and X = Ge, Si. For example, the materials Ca₃Sc₂Si₃O₁₂:Ce³⁺ and Ca₃Ga₂Ge₃O₁₂:Eu³⁺ have been studied. The materials Sr₃Y₂Ge₃O₁₂ (SYGO) phosphors have excellent photoluminescence properties by doping with the Eu³⁺ ions because Si and Ge belong to the same elemental group in the periodic table. However, SYGO: Eu³⁺ materials have not been mentioned much. In particular, there have not been detailed surveys on the influence of manufacturing methods on the properties of materials, in order to be

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used as phosphor for White LED. Thus, we selected SYGO as the host material for doping Eu³⁺ ions. We researched and produced SYGO:Eu³⁺ materials using three different processes in this report, including the co-precipitation, the solid-state reaction, and the solgel method. The phosphors have been produced, and their structure and photoluminescence properties have been studied. The phosphors were triggered with 395 nm light and emitted red light with a peak of 612 nm. However, the sol-gel method has the most monophasic structure, resulting in a higher luminescence intensity than the two co-precipitation and the solid-state reaction method.

2. Experiment

2.1. Materials and Preparation Method

All samples were made with high-purity raw materials (Sigma-Aldrich Co.) and starting main materials according to the following as strontium nitrate $(Sr(NO_3)_2)$: 3 mmol, yttrium nitrate hexahydrate $(Y(NO_3)_3.6H_2O)$: 1.95 mmol, germanium oxide (GeO₂): 3 mmol, 0.05 mmol Eu(NO₃)₃. Citric acid (HCO(COOH)(CH₂COOH)₂), and polyethylene glycol (PEG) ((C₂H₄O)n are used in the synthesis by sol-gel method with the role of network bonds and adjusting the viscosity of the solution.

Samples prepared by the solid-state reaction method were synthesized by adding all the ingredients and grinding them in an agate mortar for about 30 minutes with ethanol. The samples are dried at the conditions of 120 $^{\circ}$ C for 24 h. The dried powder is heated at 1200 $^{\circ}$ C for 8 h in the air. The process of synthesizing samples by solid-state reaction method is depicted in Fig. 1.

The process of synthesizing materials by the coprecipitation method: solution I included GeO₂ in dilute nitric acid to obtain a germanium nitrate solution. Solution II consisted of strontium nitrate, yttrium nitrate, and europium nitrate in 10 ml of DI water. After the two solutions are completely dissolved, mix solution I into solution II and stir for 30 minutes. Next, add 3 ml of NH₃OH to precipitate and stir for about 1 h to homogenize the solutions. Finally, filter and wash with distilled water or ethanol to get SYGO: Eu³⁺ powder. The samples are dried at 120 °C for 24 h. The powder is annealed at 1200 °C for 8 h in the air. The process of synthesizing samples by coprecipitation method is described in Fig. 2.

For sample SYGO: Eu^{3+} prepared by the sol-gel method: The first stir solution I and solution II for 30 minutes to obtain a homogeneous mixture. Then, 16 mol of citric acid and PEG were added and stirred for 1 h. Then, heat the stirrer at 85 °C for about 2 h to obtain a completely homogeneous solution. The next step is to open the cap, remove the magnet from the solution mixture, and leave the solution at the same temperature for 8 h. The solution evaporates slowly,

giving a homogeneous brown wet gel. Dry the prepared samples at the conditions of 120 °C for 24h. The powder is sintered at 1200 °C for 8 h in the air. The process of synthesizing samples by sol-gel method is shown in Fig. 3.



Fig. 1. Diagram for synthesizing SYGO:0.05%Eu³⁺ by solid-state reaction method







Fig. 3. Diagram for synthesizing SYGO:0.05%Eu³⁺ by sol-gel method

All samples fabricated by different methods were calcined at 1200 °C, for 8 h to ensure good crystal quality to clarify the influence of the synthesis on material properties.

2.2. Measurements and Characterization

The crystalline structures of the SYGO:Eu³⁺ nanoparticles were characterized by X-ray diffraction (XRD, D8 Advance, Bruker, Germany). The microstructure and chemical composition of the nanoparticles were determined by field emission scanning electron microscopy (JEOL, JSM7600F, JEOL Techniques, Tokyo, Japan) and energy dispersive X-ray spectroscopy (EDS, Gatan, UK). Room temperature photoluminescence (PL) tests were performed under excitation wavelength of 325 nm. NANO LOG spectrofluorometer (Horiba, USA) equipped with 450 W Xe arc lamp and double excitation monochromators was used. The PL spectra were recorded automatically during the measurements. All samples were recorded with a PMS-80 UV-VISNEAR IR spectra analysis system of SYGO:Eu³⁺.

3. Results and Discussion

3.1. Structure Properties of SYGO: Eu³⁺ Phosphor

X-ray diffraction was used to verify the phase purity of the 5% mol Eu^{3+} -doped SYGO phosphor produced using three different procedures (XRD). Fig. 4a shows the X-ray diffraction pattern of the SYGO: Eu^{3+} phosphor alongside the conventional version. The diffraction peaks in the SYGO: Eu^{3+} sample made by the co-precipitation method (symbol (3)) have an ambiguous intensity. In addition, there exist unusual GeO₂ and SrGe₂O₅ phases. The intensity of almost all of the unusual diffraction peaks is higher than that of the SYGO phase.

Due to the existence of OH⁻ radicals such as $Sr(OH)_2$, $Y(OH)_3$, and $Ge(OH)_4$, salts $Sr(NO_3)_2$, $Y(NO_3)_3$, and $Ge(NO_3)_4$ are simple to generate hydroxide precipitates with this approach when producing precipitates with NH₃OH. Under high-temperature conditions, these chemicals will react with one another. We anticipate the following reaction:

 $4Sr(OH)_2 + 2Y(OH)_3 + 6Ge(OH)_4 = Sr_3Y_2Ge_3O_{12} + GeO_2 + SrGe_2O_5 + 19H_2O$

This indicates that it matches the XRD spectrum of the sample generated using the co-precipitation process perfectly. In addition to the diffraction peaks of the SYGO host lattice, we witness the emergence of an undesirable diffraction phase dubbed GeO₂ in the sample generated via the solid phase reaction approach (symbol (2)). Since interaction between oxides is so large, unexpected diffraction phases are easily formed. In another way, the samples of SYGO: Eu^{3+} materials were synthesized by solid phase reaction method, the initial precursors are mostly nitrate salts with only GeO₂ material. At 1200 °C, which is a bit higher than the melting temperature of GeO₂, the X-ray results show that the SYGO and GeO₂ phase appears, which is completely reasonable.



Fig. 4. a) XRD pattern of the sample SYGO: Eu^{3+} generated using several methods (1) sol-gel, (2) solid-state reaction, (3) co-precipitation; b) structural crystals of the SYGO host lattice.



Fig. 5. A schematic representation of the sol-gel method's structural evolution.

The XRD model of the sol-gel-synthesized SYGO host lattice (symbol (1)) was found to be similar to the standard tag model **JCPDS #85–2410**, indicating that the sample was formed in a single phase. Included are the lattice faces 400, 420, 422, 431, 521, 532, 444, 640, 642, 800, 840, 842, 664, 1040, 1042, 880, which correspond to the 2theta angle at 27.2, 30.5, 33.6, 34.9, 37.6, 42.5, 48.1, 50.2, 52.3, 56.2, 65.3, 67.2, 78.7, 80.3, 83.5. The garnet's pure cubic crystalline phase with a space group of Ia3d is responsible for all diffraction peaks (230).

The sol-gel method can be used to produce the crystal structure development process, as shown in Fig. 5. In this, M is the symbol for the compound's element, and O is the oxygen.

For the synthesis of SYGO by sol-gel method, the precursor materials are converted into nitrate salt solution to participate in hydrolysis, condensation and cross-linking (Fig. 5). Finally, we heat the gel to form the product.

Using the *VESTA* software [6], the crystal structure of SYGO is displayed in Fig. 4b. The ions Ge^{4+} and Y^{3+} are symmetrically bound to the adjacent oxygen numbers 4 and 6, respectively. The opposite is true for the ion Sr^{2+} [7]. It is linked to the eight oxygens in the immediate vicinity as well as coordination. Asymmetry exists in this position.

The results of the XRD survey revealed that the sol-gel samples were pure and did not have any unusual diffraction peaks. The sol-gel process creates materials with a homogeneous structure, resulting in a high-purity product.

3.2. Luminescence Properties of SYGO: Eu3+ Phosphor

Excitation spectra of SYGO: Eu^{3+} phosphor synthesized by three methods for monitoring the ${}^{5}D_{0}-{}^{7}F_{2}$ emission of Eu^{3+} with an emission wavelength of $\lambda_{ex} = 612$ nm in the wavelength range of 250-500 nm are shown in Fig. 6. It shows a band of very high intensity, with a peak intensity of around ~250 nm, assigned to the charge transfer band (CTB) originating from $O^{2-} \rightarrow Eu^{3+}$. According to Garcia-Hipolito *et al.* [8], the charge transition is usually the strongest excitation mechanism, usually occurring between 250 nm and 300 nm and originating from the charge transfer from O^{2-} to Eu^{3+} ions. Other lower intensity excitation peaks are observed at 320, 365, 380, 395, 414, and 465 nm, which are attributed to the f-f transitions from the ⁷F₀ ground state to the ⁵H₃, ⁵D₄, ⁵G₄, ⁵L₆, ⁵D₃, and ⁵D₂ levels, respectively.

Fig. 6. shows the PL emission spectrum obtained with wavelengths from 500-700 nm of SYGO:Eu³⁺ phosphor synthesized by three different methods with an excitation wavelength $\lambda_{ex} = 395$ nm. The luminescence spectra of all SYGO: 0.05Eu³⁺ phosphor synthesized by three different methods show bands at 590, 612, 625, and 705 nm, with the red emission centered at 612 nm being the most prominent. On the basis of other reports [9], these bands at 590, 612, 625, and 705 nm are assigned to the ⁵D₀ \rightarrow ⁷F_J (*J* = 1,2,3,4) transitions of the Eu³⁺ ion, respectively.

The luminescence spectrum of this ion is slightly influenced by the surrounding ligands of the matrix since the electron transition of Eu^{3+} involves only the redistribution of electrons in the inner 4f subshell. The peak is centered at 590 nm, which corresponds to the ${}^{5}D_{0} \rightarrow {}^{7}F_{1}$ transition and occurs via a magnetic dipole, indicating that the Eu^{3+} ion is in a low symmetry medium. Generally, it does not change with the crystal field strength around Eu^{3+} [10].

Other peaks, were observed at 625 and 705 nm, corresponding to the transition from 5D_0 to 7F_3 and 7F_4 levels, respectively. The intensity ratio of the ${}^5D_0 \rightarrow {}^7F_2$ transition (612 nm) and the ${}^5D_0 \rightarrow {}^7F_1$ transition (590 nm) is often called the asymmetric ratio (= I_{612}/I_{590}), which is calculated as 5.8 (for samples made by the sol-gel method). The fluorescence intensity ratio allows one to measure the degree of distortion from the inverse symmetry of the local environment of the Eu³⁺ ion in the background lattice.



Fig. 6. Excitation spectrum (250-500 nm) and emission spectrum (500-750 nm) of SYGO: 0.03Eu³⁺ sample synthesized by different methods

Thus, the large asymmetric scale value indicates that the strong electric field has low symmetry at the Eu³⁺ ions. The 4f – 4f transition and the CTB are the two forms of excitation of trivalent lanthanum ions in the crystal. The 4f orbital is shielded by the two outermost layers, $5p^6$ and $6s^2$. Therefore, the influence of the lattice crystal on the optical transition in the 4fn configuration is small, and the 4f – 4f transitions are sharp lines. On the other hand, the CTB absorption exhibits a wider band and higher intensity than that of the 4f – 4f transition [11].

The fluorescence result demonstrates that the emission of the SYGO: Eu^{3+} sample made by the solgel synthesis has a stronger luminescence than the other two syntheses way (solid-state reaction and coprecipitation).

3.3. Properties of Surface Morphology, Elemental Composition, and Chemical Bonding in SYGO:Eu3+ Phosphor

Fig. 7. FE-SEM images of 0.05Eu³⁺ doped SYGO samples manufactured using various procedures demonstrate the shape and size distribution. SYGO: Eu³⁺ samples synthesized by the co-precipitation method (Fig. 7a) have a clear grain boundary formation and a size distribution of about 0.5-2 µm; samples synthesized by the sol-gel method (Fig. 7b) have a smaller particle size than the coprecipitation method but a size distribution of about 100 nm. Finally, solid phase reaction samples (Fig. 7c) are particles with no discernible form and inhomogeneous diameters ranging from 3 to 5 µm. Through X-ray, PL-PLE and FESEM analysis, it was shown that the SYGO:0.05Eu³⁺ prepared by sol-gel method gives the single-phase crystal structure. Higher fluorescence intensity and and fine powders uniformly smooth with sharp grain boundaries compared to other synthesis methods. From the above comments, we only investigated the composition of chemical elements in the sample synthesized by solgel method. Fig. 7d shows the typical EDX spectrum of the SYGO: 0.05Eu³⁺ sample generated by the solgel method. Elemental mapping samples revealed no other ions in the manufactured samples. FTIR spectroscopy was used to examine the chemical bonding feature of sample SYGO:Eu³⁺. Fig. 8 shows the FTIR spectrum at 450-4000 cm⁻¹ for the sample SYGO: 0.05Eu³⁺ generated using several procedures (co-precipitation, solid phase reaction, sol-gel method). The significant absorption bands at 830 and 690 cm⁻¹ are thought to be attributable to the asymmetric oscillation of the Ge-O bond. Alternatively, the steep peaks between 450 and 680 cm⁻¹ are attributable to the elongation of the Y-O link in the octahedral coordination state (YO₆) [12]. The strong peaks at 515 and 482 cm⁻¹ correspond to the Ge-O-Ge bond's distinctive vibrational modes. This result is quite consistent with previous publications [13].

The energy level diagram for the host SYGO and the luminescence participation process of Eu^{3+} ions are shown in Fig. 9. The electrons of the Eu^{3+} ion go from the ⁵D₀ state to the ⁷F_n level (n = 0, 1, 2, ...) of the Eu^{3+} ion with a 395 nm excitation. The Eu^{3+} ion's electrons then recombine with the hole at energies less than ⁵F_n (n=1-6), resulting in light [14].

To evaluate the applicability of phosphor, we have selected the SYGO:0.05%Eu3+ sample which was synthesized by the sol-gel method and covered the near-UV chip led. In the estimated SYGO: Eu3+ sample, the x and y coordinates are 0.65 and 0.35, respectively. In the estimated SYGO: Eu³⁺ sample, the x and y coordinates are 0.65 and 0.35, respectively. In the estimated SYGO: Eu³⁺ sample, the x and y coordinates are 0.65 and 0.35, respectively. Fig. 10. depicts the graphical depiction, with the point represented by a "dot". The CIE index of the SYGO: Eu³⁺ material is clearly in the red area, as evidenced by the symbol location in Fig. 6. As a result, it's possible that the SYGO: Eu³⁺ fluorescent powder made via the sol-gel process could be a red-emitting material.



Fig. 7. FE-SEM image of sample SYGO: 0.05Eu³⁺ synthesized by different methods: a) co-precipitation, b) solgel, c) solid phase reaction, and d) EDX spectroscopy of the SYGO: 0.05Eu³⁺ prepared by sol-gel method.



Fig. 8. FTIR spectrum of sample SYGO: Eu^{3+} synthesized by different methods



Fig. 9. Energy level diagram of the Eu^{3+} ion in the SYGO host lattice



Fig. 10. CIE color coordinate diagram of doped Eu³⁺ ions in the SYGO host lattice.

4. Conclusion

Three different methods are used to synthesize Eu^{3+} ions doped in the SYGO matrix in the air. In particular, the SYGO: Eu^{3+} material fabricated by the sol-gel method has a purer crystalline phase than the one made by co-precipitation and solid-phase reaction. The PL studies have shown the formation of emission peaks at 590, 612, 625, and 705 nm under the excitation wavelength of 395 nm, corresponding to the ${}^{5}D_{0} \rightarrow {}^{7}F_{J}$ transitions (J = 1, 2, 3, 4) due to Eu^{3+} ions. The color coordinates of the SYGO: Eu^{3+} sample prepared by the sol-gel method are x = 0.65 and y = 0.35, in the red-emitting range. Phosphor prepared by the sol-gel method has the potential to be used as a solid lighting phosphor for White LEDs.

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