1919

The effects of silica-coated Y₂O₂S:Eu³⁺ red phosphor on the lighting properties of the light-emitting diode

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ABSTRACT

The red phosphor Y₂O₂S:Eu³⁺ coated with silica (SiO₂) nanocomposite was synthesized using the sol-gel method with dip-coating technique. The purpose of coating the poly (methyl methacrylate) (PMMA)-SiO₂ composite on Y₂O₂S:Eu³⁺ phosphor's surface is to protect the phosphor and improve its scattering ability. The three primary ingredients of coating composition include methyl methacrylate (MMA) monomer, tetraethyl orthosilicate (TEOS), and SiO₂ nanoparticles. Via Mie scattering theory, the scattering of SiO₂ is examined, which primarily determines the scattering of PMMA-SiO₂-coated Y₂O₂S:Eu³⁺. The larger particles of SiO₂ in the coating composite leads to better scattering properties. When being applied in the dual-film remote phosphor configuration of a LED, SiO₂@Y₂O₂S:Eu³⁺ considerably enhances the CRI and the color quality scale (CQS). The highest CRI and CQS can be observed at approximately 85 and 74 with 23 %wt. and 26 %wt. the concentration of SiO₂@Y₂O₂S:Eu³⁺, respectively. Neverthless, the illuminating beam of the package gradually declines as the concentration of SiO₂@Y₂O₂S:Eu³⁺ go up, which might be ascribed to excessive scattering occurrences in the double-layer remote package.

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1. INTRODUCTION

Phosphor materials have been utilized in fabricating white light-emitting diodes (WLEDs) and have drawn significant researchers' attention and manufacturers in this illumination aspect. The benefits of WLED using phosphor conversion technology can be listed as durability, energy savings, affordable maintenance, and enhanced safety [1], [2]. Thus, WLED is more favorable to advance illuminating application than the conventional halogen or incandescent lights. Commonly, a criterion white LED is produced with a GaN-based LED chip that emits blue light employing phosphor YAG:Ce emits yellow light. Nonetheless, several studies presented major drawbacks due to two-color mixing of the conventional structure combining a blue-color LED die and a film of phosphor that emits yellow light, including chromatic shift and inferior CRI (about 60–70) [3]-[5]. Generally, the solution is to investigate white LEDs constitutive from fundamental colors of blue, red, and green (RGB). A white LED can be made by precoating the phosphor combination of RGB phosphors above a ultraviolet (UV) LED die, or phosphors emit green and red light above a blue-color LED chip [6]-[8]. The favorable RGB phosphors for tricolor close-UV Indium gallium nitride-light-emitting diode (InGaN-LED) chips were Y₂O₂S:Eu³⁺, ZnS, and BaMgAl₁₀O₁₇Eu2⁺ [9], [10]. Unfortunately, the red phosphor Y₂O₂S:Eu³⁺ exhibits lower photoluminescence (PL) intensity than the other green and blue ones, leading to the need for a

1920 □ ISSN: 2302-9285

combination of phosphor with a huge number of red phosphors for proper hue rendering performance. Besides, due to its instability, the endurance of $Y_2O_2S:Eu^{3+}$ under close-UV irradiation is deficient. It is now advised that the challenges with red phosphor materials should be resolved for WLEDs to improve [11]-[13]. Several efforts were made for the purpose of addressing the limitations of the $Y_2O_2S:Eu^{3+}$ red phosphor. When several alkaline earth sulfides were combined with rare-earth ion activators, this combination outperformed $Y_2O_2S:Eu^{3+}$ red phosphors [14]-[16]. However, because of the inefficient moisture resistance and instability to other atmospheric elements, the usage of alkaline earth sulfides has been restricted. The results are still insufficient, although several studies are being conducted to improve the efficacy and resistance features of alkaline earth sulfides by modifying their composition and fabrication processes. For display devices and LEDs, phosphor particles are frequently covered using oxides including silica, Y_2O_3 , ZnO, MgO, and organic polymers. Phosphor adherence to glass substrates has improved thanks to coatings, which also offers the phosphor great moisture resistance and durability, as well as protects the phosphor from being degraded when affected by UV irradiation [17]-[19].

To minimize the PL intensity loss, it is crucial to get a transparent coating with a specific amount uniformly covering the surface of each phosphor particle. About the stability, a layer-like uniform layout might be preferred over an island-like non-uniform covering. As a film-like uniform covering could be performed with littler grains having even size, it is recommended to use monodispersed nanoparticles that do not present aggregate formation as an alternative phosphor covering. We employed a dip-covering procedure with a sol-gel procedure to cover silica nanoparticles on the phosphor surface. Then, the poly(methylmethacrylate) (PMMA)-silica nanocomposite can be used to cover the phosphor surface by reacting methylmethacrylate (MMA) monomer with tetraethyl orthosilicate (TEOS). The Mie-scattering was applied to carry out the scattering analysis of the SiO₂ particles, which also determined the scattering effects of the coted red phosphor on the light properties of the WLED packages. The impacts of SiO₂@Y₂O₂S:Eu³⁺ on the double layer phosphor configuration are demonstrated via the color rendering metrics and luminous features.

2. METHOD

2.1. Materials and procedures

The Stober sol-gel process was used to create nearly mono-disperse and spherical silica particles. The experiment lasted one hour and was conducted at various temperatures. The quantities concerning reactants (TEOS and water), catalyst (ammonium hydroxide), temperature, and reaction media all influence the diameter of silica nanoparticles. Particle size decreased as TEOS and NH₄OH concentrations decreased while water concentrations increased. When the particle diameter of SiO₂ was over 25 nm, virtually monodisperse and spherical nanoparticles were generated. Meanwhile, aggregation formation was seen with SiO₂ nanoparticles smaller than 25 mm. Thus, we have to modify the temperature and medium of the reaction to prevent unwanted aggregate formation. The MMA monomer was then introduced to the reaction with TEOS to create an even coating film above the Y₂O₂S:Eu³⁺red phosphor. A high-resolution transference electron microscope (Japan) was used to examine the geometries of SiO₂ nanoparticles and Y₂O₂S:Eu³⁺ phosphors before and after coating. In addition, the median magnitude of a particle in each specimen was determined by analyzing 10 TEM pictures of 10 different specimens with image analysis software (Korea). An FTIR spectrometer-Fourier transform infrared spectrometer (USA) and an EDS-energy dispersive spectroscopy (England) was used to validate phosphor with PMMA-silica surface coating. The photoluminescence (PL) can be measured at 30 °C using a monochromatic and photomultiplier detector (Korea). Recording all of the PL spectra was given at 1 nm bandwidths at the emitting slit. Five samples of each phosphor were prepared for the PL experiment. After testing in duplicate, the outcomes of the phosphors were averaged. Variations in cathode luminescence (CL) with aging period were explored for the long-term steadiness assessment of the phosphors using a bombardment 10 kV beam of electrons with a mean current density of 45 µA/cm² for 30 minutes. Furthermore, for the tests of heat stabilization and moisture resistance of the phosphor specimens with and without PMMA-silica coating, the phosphor specimens were aged in a heat-controlled humidity chamber (Korea) at 100 °C and 80% relative humidity, followed by the test on PL strength changing with the aging period.

2.2. Simulation

As shown Figure 1(a) and Figure 1(b) presents in detail the WLED simulation with a double-layer remote phosphor design. The use of the LightTools 9.0 illumination design software and Mie scattering theory in this work allows simply simulating WLEDs with dual-layer phosphor structures by analyzing phosphor particle scattering [20], [21]. Then, based on the scattering findings, we can determine the impact of the SiO₂@Y₂O₂S:Eu³⁺ phosphor on the optic properties of the double-layer phosphor WLED packages at high correlated temperatures of 7700 K - 8500 K. For the double-layer remote phosphor configuration of WLEDs to

work properly, the SiO₂@Y₂O₂S:Eu³⁺ film is put over the yellow YAG:Ce³⁺ one, as shown Figure 1(c) and Figure 1(d). As a result, the phosphor configuration of WLEDs includes the SiO₂@Y₂O₂S:Eu³⁺ phosphor, the yellow phosphor YAG:Ce³⁺, and silicone glues.

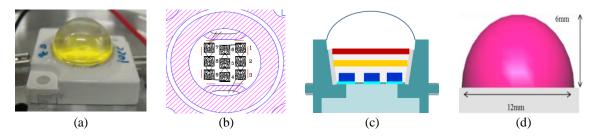


Figure 1. Picture of WLEDs configuration (a) the WLED device, (b) binding graph, (c) illustration of pc-WLEDs model, and (d) simulation of WLEDs utilizing LightTools commercial program

2.3. Scattering computation

It was observed that the particle size of SiO₂ changed depending on the reaction temperature. Particularly, when increasing the reaction temperature, the particles' magnitude declined before stabilizing. At the reaction temperature of about 30 °C, monodisperse SiO₂ nanoparticles with 50 nm particle size were formed. However, when that temperature increased to over 80 °C, the particle magnitude fell down to roughly 10 nm. Bigger particles (at least 30 nm) generated nearly monodisperse and spherical nanoparticles. It is worth noting that aggregates formed when primary silica nanoparticles smaller than 20 nm were present. There are various plausible explanations for these findings. For example, additional particles are formed when smaller particles are generated. Because tiny particles have a greater outside tension than huge particles, they mix faster to create a more solid surface. As a result, primary silica particles with sizes smaller than around 20 nm produced a network formation due to aggregation, whereas spherical particles with diameters higher than 20 nm produced a spherical structure without aggregation. Increasing the water concentration lowered the diameter of silica nanoparticles from 50 to 10 nm at a reaction heat 30 °C-80 °C. When particle diameters were smaller than roughly 20 nm, however, aggregates containing main silica nanoparticles developed. The data demonstrate that greater reaction temperature can get the size of SiO₂ nanoparticles decreased even further, but solely changing the reaction temperature would not solve the aggregation formation problems.

The scattering properties of the red phosphor $Y_2O_2S:Eu^{3+}$ are probably influenced when the PMMA-SiO₂ nanocomposite covers its surface. The changes are possibly caused by the scattering ability of the SiO₂ particles, which fluctuates owing to the particle size modification. In this study, the Mie-scattering theory is utilized to calculate and analyze the dispersing properties of SiO₂ particles. Relying on Mie-scattering theory [22]-[25], formulas (1)-(3) can be used to compute the dispersing factor $\mu_{sca}(\lambda)$, anisotropy factor $g(\lambda)$, and decreased dispersing factor $\delta_{sca}(\lambda)$:

$$\mu_{sca} = \int N(r)C_{sca}(\lambda, r)dr \tag{1}$$

$$g(\lambda) = 2\pi \int_{-1}^{1} p(\theta, \lambda, r) f(r) \cos\theta d\cos\theta dr$$
 (2)

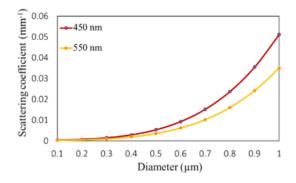
$$\delta_{sca} = \mu_{sca}(1 - g) \tag{3}$$

While N(r) represents the dispensation density of diffusional particles (mm³), C_{sca} indicates the dispersing cross-sections (mm^2), $p(\theta, \lambda, r)$, λ , and r are the phase function, the illuminating wavelength (nm), and radius of diffusional particles (μm), respectively. θ means the dispersing angle (°C), and f(r) displays the size dispensation function of the diffusional particles in the phosphor package.

As shown Figure 2 and Figure 3, the increase in diameter of SiO₂ particles boosted the scattering properties. Moreover, the results at the blue light wavelength (450 nm) are higher than that of the yellow one (550 nm), implying that the blue-light absorption could be better. When enhancing the absorption ability of Y₂O₂S:Eu³⁺ phosphor with PMMA-SiO₂ coating, the red-light proportion converted from the scattered blue lights is likely to increase. It contributes to the improvement in chromaticity of WLED dual-layer distant phosphor packet. Additionally, the scattering enhancement that SiO₂@Y₂O₂S:Eu³⁺ red phosphor brings to the

1922
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phosphor structure has significant effects on the overall luminous output of the white LED configuration, which will be discussed in the next section.



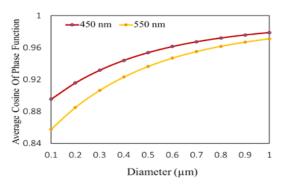


Figure 2. Dispersing factors of SiO_2 particles at 450 nm and 550 nm

Figure 3. The phase function of SiO_2 particles at 450 nm and 550 nm

3. RESULTS AND DISCUSSION

The influences of the SiO₂-coated $Y_2O_2S:Eu^{3+}$ on the lighting properties of the WLED employing two remote phosphor layer designs are presented and discussed in this part. Judging the experimental results, the concentration of $SiO_2@Y_2O_2S:Eu^{3+}$ is the main force of the changes in optical performance. The relation between the concentrations of the yellow YAG:Ce³⁺ and SiO₂-coated Y₂O₂S:Eu³⁺ phosphors will be demonstrated as shown in Figure 4. The remarkable reduction of the yellow-color phosphor weight percentage, at both correlated color temperatures (CCTs) 7700 K and 8500 K. The reduction in yellow phosphor YAG:Ce³⁺ concentration is the result of the scattering enhancement when the SiO₂@Y₂O₂S:Eu³⁺ phosphor is employed. This has two primary benefits to the lighting features of WLEDs, which are the color temperatures consistency and the encourage the reduction of back-scattering that causes light loss in the LED structure. Hence, coating SiO₂ composite on the red phosphor Y₂O₂S:Eu³⁺ enhances both the stability and the scattering of the core phosphor, resulting in improvement of the LED lights.

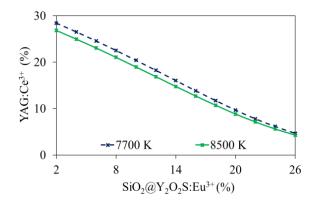
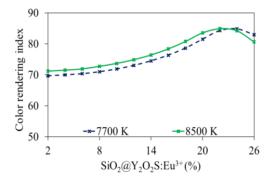


Figure 4. The shift of phosphor concentration occurring to maintain the median CCTs

In terms of the chromaticity of the double-film distant phosphor structure, the CRI and color quality scale (CQS) are investigated. CRI is among the parameters most commonly utilized in the chromatic fidelity test. We can gain a high CRI via enhancing the red energy in the overall spectral emission of the white light. The CRI of the WLED in connection with the concentration of SiO₂@Y₂O₂S:Eu³⁺ is presented as shown in Figure 5. As shown in the picture, the presence of the coated red phosphor is beneficial to the CRI. At the lowest applied concentration (2 %wt), it is possible for the WLED to attain the CRI of 70. Subsequently, increasing the amount of SiO₂@Y₂O₂S:Eu³⁺ content gets the CRI of the structure heightened. The CRI peak can be observed at about 85 with 23 %wt SiO₂@Y₂O₂S:Eu³⁺ in the package. As the concentration surpasses 23 %wt the CRI slightly decreases to 80, which might be caused by the excessive red-light component

leading to color-distribution imbalance. The scattering enhancement offered from the application of $SiO_2@Y_2O_2S:Eu^{3+}$ phosphor could be explained for a better CRI. As there are more blue lights scattered, it heightens the change of light mixing between the blue and yellow lights, and thus stimulates the uniformity of the generated light. Moreover, the red phosphors induce the blue-light absorption and convert those blue lights into red lights which is essential to the CRI improvement. Not only the CRI is promoted but also the CQS. National Institute of Standards and Technology's (NIST's) researchers developed CQS in the form of a novel hue rendering metric to overcome the limits of traditional CRI, such as a limited tested color sample. The CQS is believed to be better than the CRI since it evaluates a light source with 15 new color samples; plus, it examines the color coordinate and human visual preference. The CQS changes with the concentration of red phosphor $SiO_2@Y_2O_2S:Eu^{3+}$ are demonstrated as shown in Figure 5. Similar to the CRI, the CQS would be proportional to the $SiO_2@Y_2O_2S:Eu^{3+}$ concentration. In particular, the higher the mass percentage in $SiO_2@Y_2O_2S:Eu^{3+}$ presents, the better the CQS becomes. The CQS peak is about 74 when adding 26 %wt to the phosphor package, see Figure 6. In brief, using the $SiO_2@Y_2O_2S:Eu^{3+}$ red phosphor has tremendous advantages to the hue output in the WLED as the scattering properties of original $Y_2O_2S:Eu^{3+}$ are enhanced with PMMA-SiO₂ coating.



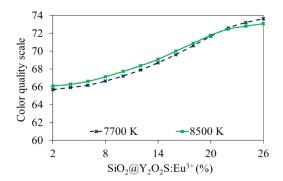


Figure 5. The CRI of WLEDs and SiO₂@Y₂O₂S:Eu³⁺ concentration

Figure 6. The hue standard scale of WLEDs and SiO₂@Y₂O₂S:Eu³⁺ concentration

Last but not least, the luminous efficiency of the WLED with $SiO_2@Y_2O_2S:Eu^{3+}$ layer is demonstrated. In contrast to the CRI and the CQS, the lumen output significantly decreases when the content of the $SiO_2@Y_2O_2S:Eu^{3+}$ increases, see Figure 7. At 23-26 %wt. the luminous flux is around 400 lm, which is reduced by approximately 600 lm, compared to the figure at 2 %wt $SiO_2@Y_2O_2S:Eu^{3+}$. Therefore, to successfully apply $SiO_2@Y_2O_2S:Eu^{3+}$ red phosphor to achieve optical-performance improvement for WLEDs, the consideration and modification of this phosphor concentration are critical.

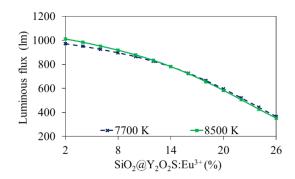


Figure 7. The illuminating beam of WLEDs and SiO₂@Y₂O₂S:Eu³⁺ concentration

4. CONCLUSION

PMMA-SiO₂ nanocomposite is coated above the exterior of red phosphor $Y_2O_2S:Eu^{3+}$ using sol-gel method with dip-coating technique followed by the reaction of MMA monomer and TEOS. The particle magnitude for nanoparticle SiO₂ changes in connection with the reaction temperature. High temperature

1924 □ ISSN: 2302-9285

 $(80~^{\circ}\text{C})$ reduced the particle size to 10~nm. The fluctuation in the size of SiO_2 particles also affects the scattering properties of the material. The bigger particle presents better scattering properties, according to the results obtained via Mie scattering theory. The impacts of $SiO_2@Y_2O_2S:Eu^{3+}$ on the lighting performances of double-layer remote phosphor structure are investigated, regarding its concentration. The growth in $SiO_2@Y_2O_2S:Eu^{3+}$ concentration significantly enhances either CRI or CQS. By using more than 20~%wt. $SiO_2@Y_2O_2S:Eu^{3+}$, peaks of CRI and CQS at approximately 85~and~74~can be accomplished, respectively. Nevertheless, the rise of $SiO_2@Y_2O_2S:Eu^{3+}$ causes a considerable reduction in the luminous output of the package.

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