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Industry-friendly Synthesis and High Saturation Threshold of a LuAG:Ce/Glass Composite Film Realizing High-brightness Laser Lighting

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ABSTRACT

Phosphors that are designed for laser-lighting are essential to possess high luminescence saturation threshold. However, the current phosphors with adequate saturation threshold have been facing several technological limitations including costly, complex and tedious synthetic approaches. Herein, we report the facile fabrication of LuAG:Ce/glass composite films using an industry-friendly blade-coating method. The resultant composite film presents a porous architecture that can facilitate light extraction and confine the laser spots. When pumped by blue laser, the composite film shows a high internal quantum efficiency of 86% and the corresponding white light source shows a high luminous efficacy of 199 lm/W. Furthermore, the composite film shows no sign of

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saturation even under the irradiation of 30.1 W blue laser; therefore, a high luminous flux of 5496 lm can be achieved. The combination of these outstanding properties and the industry-friendly fabrication method can drive the development of commercially viable phosphors for high-power laser-lighting applications.

KEYWORDS: laser lighting, LuAG:Ce, composite film; blade coating; high luminous flux

1. Introduction

The development of solid-state lighting technology has led to several advancements in the area of high-luminance and high-directionality applications [1-3]. Despite the considerable success of white light-emitting diodes (wLEDs) in lighting and displays, they are unfortunately unsuitable for high-luminance and high-directionality applications because of their high emission beam divergence (Lambertian), relatively large emission area, and the notorious "efficiency droop" problem [2, 4, 5]. In comparison, laser diodes (LDs) do not suffer from "efficiency droop"; therefore, they can maintain a high wall-plug efficiency at a high current density [4-8]. Furthermore, LDs exhibit considerably lower beam divergence than LEDs, indicating their obvious superiority for small luminaire devices. Similar to wLEDs, phosphors are commonly used as color converters for generating white light in laser pumped white lighting [1, 2]. However, the high flux density from LDs along with the related massive heat accumulation leads to new challenges for developing phosphors. For example, the conventional organic resin-based phosphor coating is inadequate for practical use in laser lighting, because of their poor stability and low thermal conductivity.

To accommodate the challenges of LDs, robust and efficient all-inorganic phosphors have become a research hotspot [3, 4, 9-14]. Zhu et al. reported a green-emitting phosphor-in-glass (PiG),

where glass was used as matrix and bonding material which successfully avoided the use of resin; however, this PiG is still inadequate for practical use because of its low saturation threshold (~0.98 W/mm^2) [15]. Subsequently, several types of PiGs were developed, which also saturated at a very low laser power/power density, probably because of the poor thermal conductivity (~1 W/m·K) of the glass matrix [2, 16-20]. To improve the saturation threshold, phosphors evolved from PiGs to ceramics and single crystals. Xu et al. developed a LuAG:Ce-based ceramic phosphor, which showed an improved saturation threshold of >24 W/mm^2 [14]. Li et al. designed a Al_2O_3/YAG:Ce composite ceramic using Al_2O_3 as the matrix; furthermore, the Al_2O_3 can act as heat sink because of its excellent thermal conductivity (~30 W/m·K) [11, 21]. This ceramic phosphor had a high saturation threshold of >50 W/mm^2. Subsequently, considerable progress has been made by Xu et al. who developed a YAG:Ce-based single crystal phosphor, which showed a high saturation threshold of >360 W/mm^2 [3]. However, despite the impressive saturation threshold, the development of ceramic and single crystal phosphors has been limited by several technical difficulties such as costly, complex, and tedious synthetic approaches (e.g., spark plasma sintering, hot pressing sintering, vacuum sintering, Czochralski method), low yield, and/or carbon contamination [3, 4, 11, 14, 18, 22-24]. Thus, it is challenging to fabricate all-inorganic phosphors with high saturation threshold using a facile and industry-friendly synthetic route.

To obtain a high saturation threshold, a phosphor for laser lighting has to satisfy the following criteria: (i) the phosphor's luminescence thermal quenching has to be small; (ii) the phosphor should be able to efficiently dissipate the generated heat; (iii) the phosphor should have a high internal quantum efficiency (IQE) to reduce the spectral-conversion loss; and (iv) the luminescence decay of the activators from the excited state to the ground state should be fast. Herein, we designed a

LuAG:Ce/glass composite film that was synthesized via a facile and industry-friendly blade-coating method. The as-prepared film inherited a high IQE and low thermal quenching from the pristine LuAG:Ce powder. Furthermore, because of its shorter distance to the heatsink (substrate), the composite film naturally possesses good thermal dissipation. The composite film shows a high saturation threshold that it can withstand the irradiation of a 30.1 W blue laser; therefore, a high luminous flux of 5496 lm was attained. With these features, the as-designed composite film may open doors toward commercially viable color converters for ultrahigh luminance laser lighting.

2. Experimental

2.1. Fabrication of the composite film

The composite films were fabricated using a blade-coating method, as shown in Figure 1. Glass powder (B_2O_3-Al_2O_3-ZnO-SiO_2 system, $T_g \approx 550$ °C, $T_s \approx 605$ °C) and 0.2 g of LuAG:Ce powder (Yantai Shield, SDL-525) were weighed and mixed at a mass ratio of Glass/LuAG:Ce = 1/4, 1/2, 3/4, and 1/1. The mixtures were dispersed in an organic vehicle of 5 ml ethyl acetate (Aladdin, CAS 141-78-6), 2 ml terpineol (Aladdin, CAS 8000-41-7), and 0.2 g ethyl cellulose (Aladdin, CAS 9004-57-3). After vigorous stirring for 4 h, a viscous ink was obtained; then it was printed on an alumina substrate (19 × 14 × 2 mm) and heated to 120 °C to remove the organics. Subsequently, the samples were annealed in a muffle furnace at various temperatures (600–900 °C). All the heat treatments were conducted in ambient air. The soaking time was 20 min, and the thickness could be controlled by the number of layers.

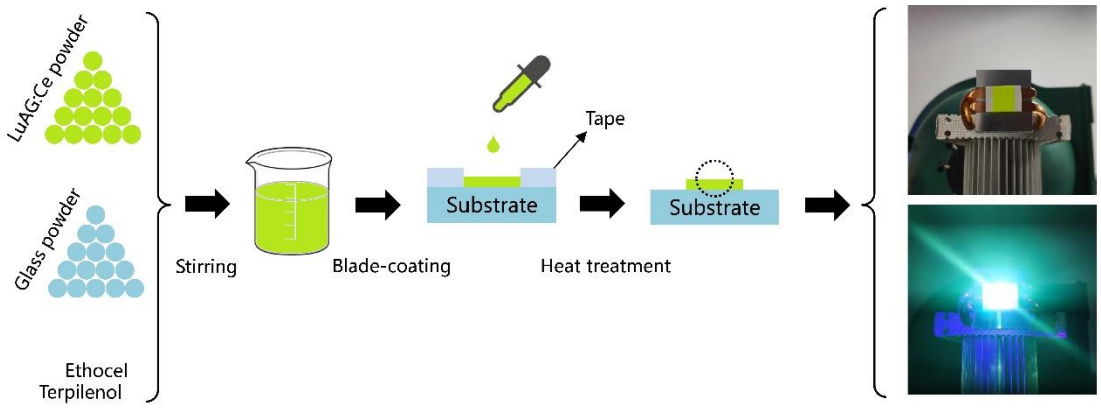

Figure. 1 Schematic of the LuAG:Ce/glass composite film production; insets are the image of a typical sample and its lighting effect (pumped by a 0.5 W blue laser). The weight ratio of glass/LuAG:Ce is 1/4, and the annealing temperature is 800 °C.

2.2. Characterization

The surface morphologies were observed using a scanning electron microscope (SEM, Hitachi, TM-3030 plus). The luminescence properties of the phosphors were measured using a spectroradiometer (HORIBA, Fluorolog-3) equipped with an integrating sphere (Labsphere, diameter = 30 cm). The laser-pumped lighting properties were evaluated using a sphere-spectroradiometer system consisting an array spectrometer (Instrument Systems, CAS-140-CT-151) with a fiber-coupled integrating sphere (Labsphere, diameter = 15 cm). The high-power laser module was constructed by integrating eight (i.e., 2 × 4 array) blue LDs (NUBM-08, 4.75 W × 8, $\lambda_{em} \approx 450$ nm, Nichia). The laser beams were focused to a spot size of ~0.17 cm^2 using a telescopic lens system focusing at the exit of the integrating sphere. The laser power was measured using a power meter (Ophir, NOVA II). The angular CCT distributions were characterized via a home-made measurement system. A fiber-coupled spectrometer (Ocean Optics, QE65000) was used as the optical sensor. A 3 W blue laser (445 nm) was used as the pumping source. The sensor angle can be dynamically adjusted by a robot arm to collect the angular dependence of emission spectra.

3. Results

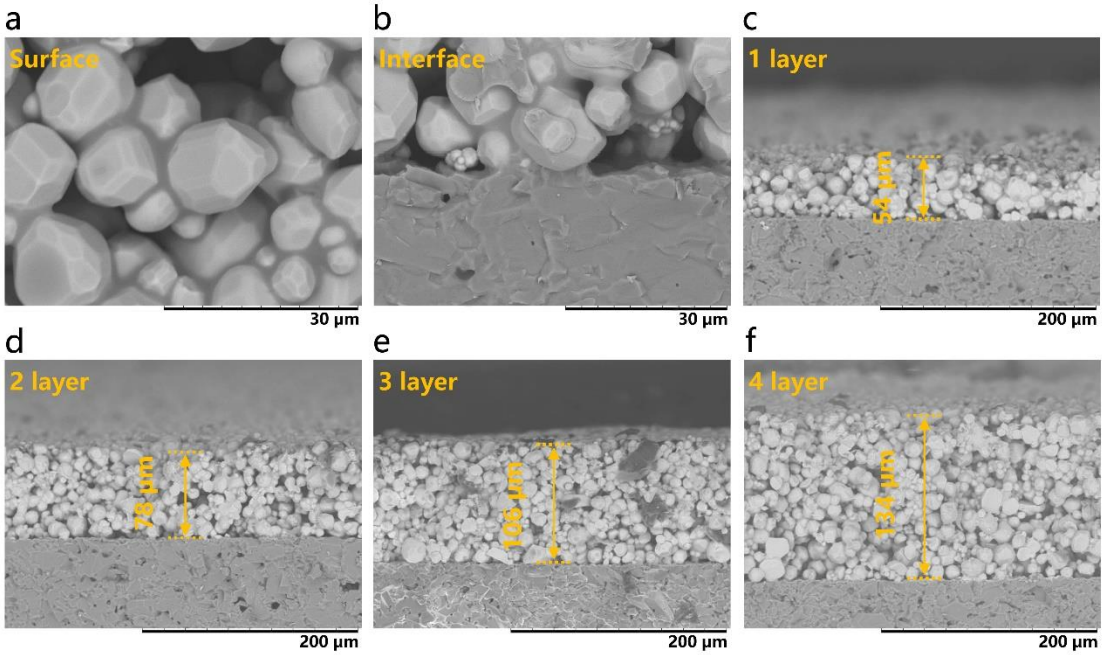

Figure. 2 SEM images of the surface (a), cross section (b), and different thickness (c-f) of the composite films. The weight ratio of glass/LuAG:Ce is 1/4, and the annealing temperature is 800 °C.

Using SEM, the morphologies of the composite films were investigated. Figure 2a,b shows the surface and interface SEM images (between film and substrate) of a typical sample (the weight ratio of glass/LuAG:Ce is 1/4; the annealing temperature is 800 °C, 1 layer, hereafter denoted as Sample-1/4-800-L1). Note that the glass powder completely melted and was well wetted on the surface of the phosphor particles. For each LuAG:Ce particle, the melted glass acted as the bonding material and heat transfer media. Furthermore, the film was tightly bonded to the substrate (Figure 2b), indicating its good adhesion and durability. By controlling the number of layers, four different thicknesses, i.e., 54, 78, 106, and 134 μm, were obtained (Figure 2c-f). Note that the film showed a porous microstructure. During the annealing process, the glass melts and contracts which can drain the residual air. However, the phosphor powder is very stable at this temperature, and it can act as a barrier that traps the air, and thus a porous microstructure can be formed. The pores and the

phosphor-to-glass interfaces can be seen as strong scattering sources in the film. As per the conventional design principles of a phosphor, a low level of light scattering is required to minimize the scattering loss [2, 25]. However, recent studies have reported that a relatively strong light scattering can effectively enhance the (blue) light extraction, facilitate the color mixing, and confine the luminescent spots [4].

Figure 3 shows the luminescence properties of the composite films. Under an excitation of 450 nm, the pristine LuAG:Ce powder presents a broad, greenish yellow (460–700 nm) emission band (Figure 3a) due to the 5d → 4f ($^2F_{7/2}$ and $^2F_{5/2}$) transition of Ce^{3+}. Generally, the film (Sample-1/4-800-L1) can inherit the luminescence properties of the LuAG:Ce powder. However, a small blue shift (623 → 619 nm) can be observed because the reabsorption between the phosphor particles is mitigated by glass. The CIE coordinates of the pristine LuAG:Ce powder and the composite film are (0.3429, 0.5815) and (0.3392, 0.5838), respectively. It is noteworthy that a weak emission peak at 695 nm can be observed. The chromium hemitrioxide is commonly used as sintering aid in the producition of corundum, therefore the peak at 695 nm is attributed to the luminescnece of Cr^{3+} in the Al_2O_3 host [26, 27]. Figure 3b shows the thermal quneching behaviors of LuAG:Ce powder and the Sample-1/4-800-L1. The large energy gap between the $5d_1$ emitting level of Ce^{3+} and the conduction band buttom of LuAG ensures a thermally robust luminescence of Ce^{3+} in LuAG host [28, 29]. Thus, the LuAG:Ce powder at 200 °C can maintain ~96% of the (integrated) relative intensity at room temperature. The composite film inherits good thermal stability of luminescence from the powder. Compared with the initial intensity at room temperature, the intensity drop of the film at 200 °C is within 5%. Figure 3c shows the effect of annealing temperature on the IQE of the Sample-1/4-800-L1. From 650°C to 800 °C, the composite film shows a high and stable IQE of

~86%, which starts to obviously drop at 850 °C because the glass could be chemically agreessive at higher temperature, induce defects and thus affect the phosphor's luminescence. Figure 3d shows the effect of the glass/LuAG:Ce mass on the IQE of Sample-800-L1. With increasing weight ratio of the glass in the PiG mixture (1/4 → 1/1), the IQE presents a slightly decreasing trend. Higher glass ratio tends to result in higher light transmittance, which could lead to more light reabsorption and thus reduce the IQE. However, the overall decrease is only ~3% (86% → 83%), which is insignificant. Based on the abovementinoned comprehensive assessment, the Sample-1/4-800 with different thicknesses was selected for evaluating the laser-pumped lighting properties.

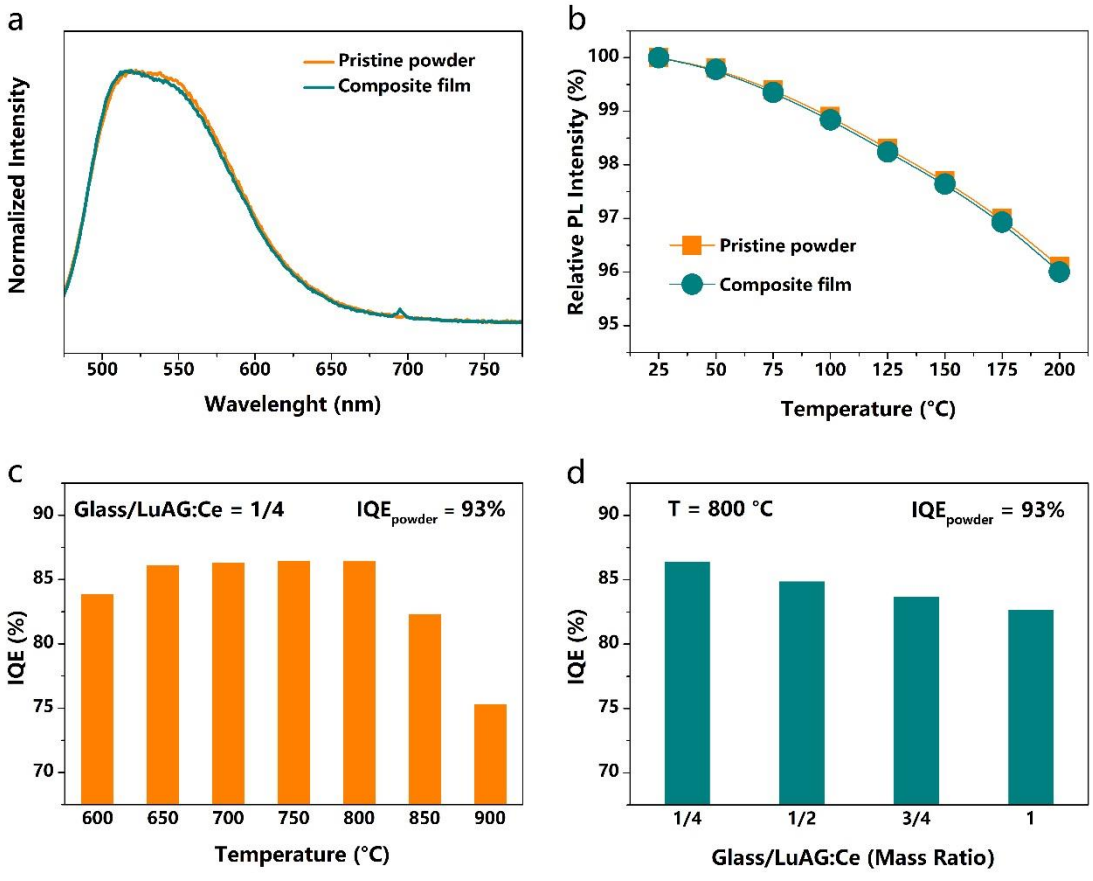

Figure. 3 (a) PL (λ_{ex} = 450 nm) spectra and (b) temperature-dependent luminescence intensity for the pristine phosphor powder and Sample-1/4-800-L1. Effects of the annealing temperature (c) and glass/LuAG:Ce mass ratio (d) on the IQE of the films.

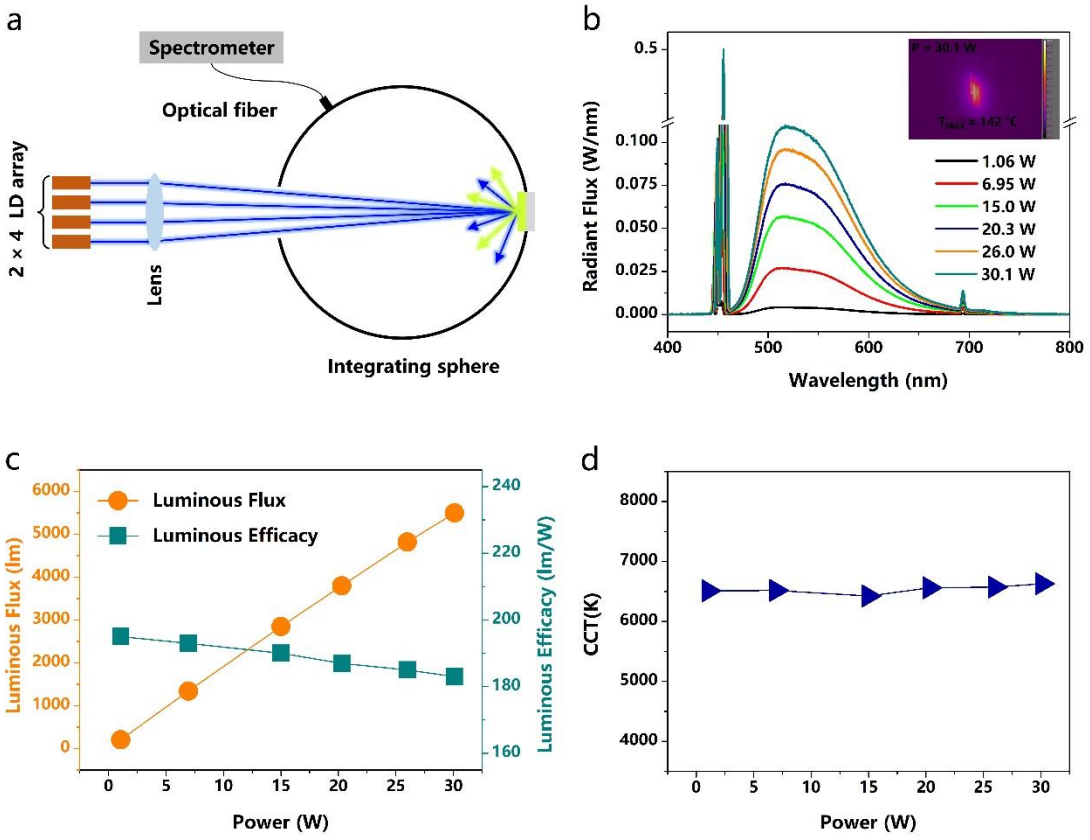

Figure. 4 (a) Schematic of the high-power laser module and the sphere-spectroradiometer system in the reflective configuration. Laser-pumped emission spectra (b), luminous flux/efficacy (c), and CCT (d) of the Sample-1/4-800-L4, with the laser power increasing from 1.06 to 30.1 W. Inset in (b) is the infrared thermal image of the film under 30.1 W laser irradiation.

Using the calibrated sphere-spectroradiometer system, the laser-pumped lighting properties of the samples were systematically evaluated in reflective configuration. Figure 4a shows the measurement setup where the the samples were placed at the sample port of the integrating sphere and illuminated by a 2 × 4 blue laser array. The laser power-dependent emission spectra and the optical properties of Sample-1/4-800-L4 were investigated, and the results are shown in Figure 4b-d. In the entire laser power range (from 1.06 to 30.1 W), the emission intensity of the sample monotonously increased with the increase in laser power showing no sign of luminescence saturation. Therefore, the luminious flux of the sample presented a continuous and linear increase. Under the irradation of 30.1 W blue laser, an high luminous flux of 5496 lm was achieved. The

luminous efficacy was relatively stable and decreased only slightly as the incident laser power increased. The luminous efficacy of the sample at 30.1 W was ~183 lm/W; thus ~92% of the initial value (~199 lm/W) was maintained. The operating temperature of the sample monotonously increased with increasing laser power and reached ~142 °C under a laser irradiation of 30.1 W (inset in Figure 4b). Thus, the corresponding thermal quenching was insignificant (~2% intensity droop). Note that the emission spectra of the sample (in Figure 4b) shows a clear red shift (511 nm → 518 nm) because the elevated temperature broadens the emission band, which increases the spectral overlap between the excitation and emission spectra. This ultimately results in a red shift in the emission spectra. With increasing laser power, the CCT is very stable with a maximum variation of 2% (6496 K → 6629 K). The decline in the luminous efficacy can increase the CCT by increasing the ratio of blue/yellow light; on the contrary, the red shift of the emission spectra would decrease the CCT. These two factors could be counteracted, which allows to achieve an invariable CCT.

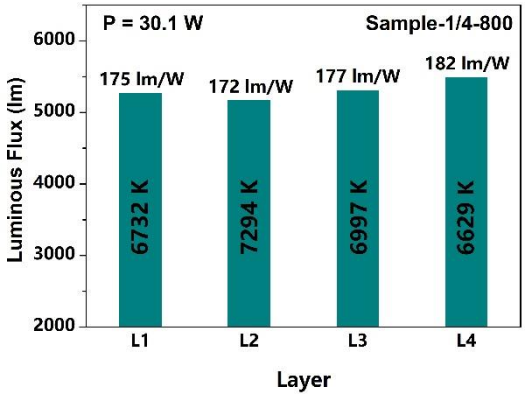

Figure 5 Effects of thickness (number of layers) on the luminous flux/efficacy of the Sample-1/4-800.

Figure 5 shows the effects of the thickness on the luminous flux/efficacy and CCT of the composite film. Recently, many studies have revealed that the thickness can significantly affect the optical properties of a phosphor film. Wei et al. reported that the luminous flux/efficacy of a YAG:Ce/glass film obviously decreases as the thickness increases [30]. Surprisingly, in this study,

the effect of thickness on the luminous flux/efficacy and CCT of the composite film was insignificant. In a thickness range from 54 to 134 μm, all the four typical samples irradiated at 30.1 W (the maximum power) show similar luminous flux/efficacy and CCT, and the corresponding maximum variations are only 320 lm (5496 lm → 5176 lm, ~6%), 10 lm/W (182 lm/W → 172 lm/W, ~5%) and 665 K (7294 K → 6629 K, ~9%), respectively. These unusual results are probably because of the following reasons. First, in previous studies, the measurements were conducted in a transmissive configuration where transmittance is one of most important properties for the phosphor samples. The thickness can directly affect the transmittance and thus significantly affect the lighting properties. In this study, the measurement was conducted in a reflective configuration such that the transmittance was not as important as that in previous studies; thus, the thickness was insignificant. Second, the glass/phosphor ratios in previous studies were relatively high (e.g., >1/1), thus yielding a high transmittance. A higher glass/phosphor ratio indicates lower porosity and weaker light scattering, which could reduce the blue light extraction. Thus, changing the thickness helped achieve a balance between the transmittance and blue light extraction, which therefore effectively affected the lighting properties of the phosphor films. In this study, the low glass/phosphor ratio (e.g., 1/4) can lead to high porosity and strong light scattering. Thus, adjusting the thickness was not necessary to obtain sufficient extraction of blue light. Consequently, the thickness can hardly affect the lighting properties of the samples. Even though the maximum laser power in this study reached as high as 30.1 W (~170 W/cm^2), it is still insufficient to saturate the composite film because of its very small thermal quenching, fast decay, and good heat dissipation. Thus, the effect of the thickness on the optical properties, especially for the saturation threshold, could be hidden. In future studies, it could be possible to saturate the samples by using a more powerful laser (e.g., >50 W) or a smaller spot

size so that the effect of the sample thickness on the lighting properties would be clarified.

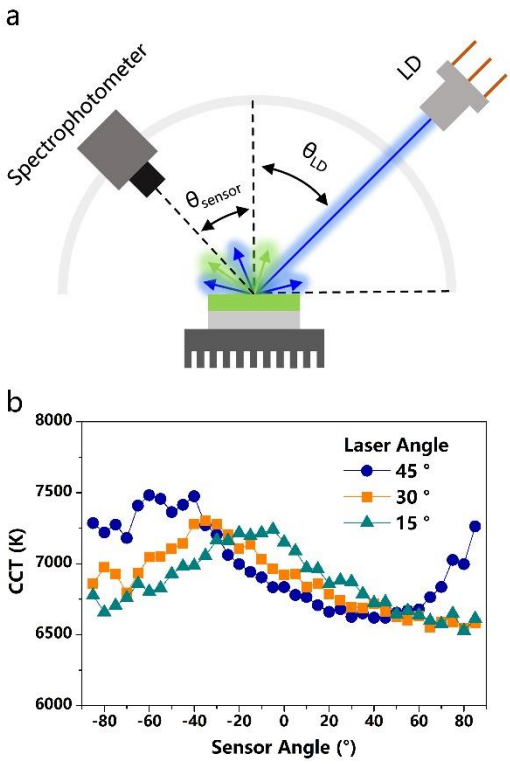

Figure. 6 (a) Schematic for measuring the angular dependence of CCT distribution. (b) Angular CCT distribution of the Sample-1/4-800-L1.

Besides the energy efficiency and average CCT, the angular color uniformity (ACU) is also one of the key parameters to evaluate the light quality of a lighting system. For a wLED, the ACU is not a problem because of its Lambertian intensity distribution of the pumping source. Whereas, ACU for a laser lighting device is generally low because the pumping laser beam is collimated or focused. To evaluate the ACU of the composite film based light source, an experimental setup was built for reflective configuration, showing in Figure 6a. During measurement, the Sample-1/4-800-L1 was fixed on a substrate. The angle of the spectrophotometer (θ_{sensor}) can be dynamically adjusted by a robot arm to collect the angular dependence of the emission spectra. The spectrophotometer rotated from 5° to 85° with increment of 5°. Three incident LD angles (θ_{LD} = 15°, 30° and 45°, respectively) were selected. The ACU results are presented in Figure 6b. At LD angles of 15°, 30°

and 45°, the corresponding highest CCT value appeared at around the sensor angle of -15°, -30° and -45°. This means there is still distribution preference for the laser light because of the surface reflection. However, the maximum CCT variations at all three LD angles are within 1000K which is acceptable. Furthermore, the center of the emission spot for composite film is whitish that no blue spot is observable (see Figure 1). Therefore, the ACU of the composite film-based lighting system is obviously superior to that of the resign-based phosphor films as well as the single crystal phosphors [21, 23, 31, 32]. This favorable ACU can be ascribed to the strong scattering originated from the porous film architecture (see Figure 2).

Conclusions

In summary, a robust and efficient LuAG:Ce/glass composite film with a porous architecture was fabricated using a facile and economical blade-coating method. Under the irradiation by a blue laser, the composite film presented a high IQE of ~86% and a high luminous efficacy of 199 lm/W. Furthermore, it could withstand a high laser power of >30.1 W (170 W/cm^2) and generate a high luminous flux of >5496 lm. Interestingly, the effect of thickness on the optical properties of the composite film was insignificant under the reflection measurement mode, which could make the synthetic route very industry friendly. With the abovementioned features, an excellent balance between the preparation costs and the laser-pumped optical properties can be successfully achieved. The composite film combined with the synthetic route may open doors towards commercially viable color converters for high-brightness laser-lighting applications.

Acknowledgements

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