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Abstract: High color rendering index (CRI) of 97 and wide correlated-color temperatures (CCTs) range of 2100–7600 K in white light-emitting diodes (WLEDs) employing the two-inch diameter of $CaAlSiN_3$: Eu^{2+} (red phosphor) and $Lu_3Al_5O_{12}$: Ce^{3+} (green phosphor) co-doped phosphor-in-glass (PiG) are demonstrated by a novel wet-type cold isostatic pressing (CIP) technique. The wet-type CIP enable to apply isostatic pressure to a powder sample in all directions to obtain the PiG with excellent optical properties, high reliability, and thermal stability. The high CRI of 97 and the wide CCTs range of 2100–7600 K achieved are mainly due to the optimized phosphor materials and the precise adjustment thickness of the PiG. Based on the best formula of two-inch diameter PiG and a unique process of wet-type CIP, we have successfully fabricated and demonstrated the good uniformity and excellent performances of a large diameter of two-inch PiG. The proposed of a large diameter of two-inch PiG with excellent performance, high reliability, and thermal stability is essential to meet the economic dimension requirement for commercial usages in high-quality WLEDs indoor lighting applications.

Index Terms: White light-emitting diodes (WLEDs), phosphor-in-glass (PiG), color rendering index (CRI), correlated-color temperature (CCT), wet-type cold isostatic pressing (CIP).

1. Introduction

Color plays as an important role in the daily life of human culture. As the rapid growth of the solid-state lighting industry, the requirement for the variety and fidelity of the color performance is more than before, especially focusing on the characteristics of color rendering index (CRI) and correlated-color temperature (CCT) [1], [2]. Many studies have shown that the effects of artificial lighting on human health and the characteristics of the light may influence human physiology and psychology [3], [4]. For instance, the CCT of the warm white light is lower than 3300 K, which can give people a comfortable feeling and suitable for houses and dormitories. The CCT of the neutral white light is between 3300 and 5300 K. Due to the soft light, neutral white light is pleasant, comfortable, quiet, and suitable for many places, such as hospitals and restaurants. However, the

CCT of the cool white light is above 5300 K, which is close to natural light and has a bright feeling. This may allow people to concentrate and is suitable for use in meeting rooms and classrooms. Therefore, in view of the various functions and environments in the real world, it is important to choose a suitable CCT of light source [5]. The CRI is also important characteristics of the light. It is a key parameter for evaluating the light quality of the white light sources that deviate from the natural sunlight and black body radiation. High CRI with full-spectrum lighting can significantly improve the appearance of the objects. Usually, the CRI is greater than 80 as a standard indoor lighting source. However, due to the improvement of the quality of life in recent years, humans have paid more attention to the quality of the lighting sources. From manufacturer point of view, the CRI greater than 90 is prefer for natural daylight sources [6]. Solid-state lighting sources such as white light-emitting diodes (WLEDs) have been widely used in the field of the general lighting due to their small dimension, low cost, long life, high efficiency, fast response speed, and acceptable color performance [7], [8].

Phosphor-converted WLED (pc-WLED) is the most appropriate architecture that can be used for small solid-state light sources with the desired emission spectra. The pc-WLEDs had provided the most cost-efficient solution in the field of the general indoor lighting [9], [10]. Recently, many studies have pointed out that the thermal stability of the phosphor-in-glass (PiG) is much higher than that of the silicon-based phosphor and can be used as a solution for high-power WLED conversion layer [11], [12], [13]. However, high CRI over 90 with a complete range of white light CCTs for the WLEDs fabricated by the PiG have not been reported in the previous study [13]. Therefore, reconstructing the characteristics of the black-body-like light sources of the PiG to achieve both wide CCTs range and high CRI used in the high-quality indoor lighting applications is necessary. Furthermore, a small diameter of 13-mm PiG for high CRI fabrication [13], which may not meet the economic dimension requirement for commercial usages. Therefore, development of a large diameter of 2-inch PiG with excellent optical properties, high reliability, and thermal stability is essential and necessary to be further investigation.

In this work, we studied to demonstrate the high CRI of 97 and the wide CCTs range of 2100–7600 K in the PiG-based WLEDs by using four various golden formulas based on only one combination of $CaAlSiN_3$: Eu^{2+} (red phosphor) and $Lu_3Al_5O_{12}$: Ce^{3+} (green phosphor). The high CRI of 97 and the wide CCTs range of 2100–7600 K achieved are mainly due to the optimized phosphor materials and the precise adjustment thickness of the PiG. The results show that the high CRI of 97 and the wide CCTs range of 2100–7600 K of the 13-mm diameter PiG in the WLEDs are obtained, which can be directly used for the development of 2-inch diameter PiG. In order to achieve a diameter of 2-inch PiG with excellent optical performance, high reliability, and thermal stability, a novel wet-type cold isostatic pressing (CIP) technique needs to be performed, which is a powder compaction technique that applies an isostatic pressure to a powder sample in all directions. Based on the best formula of 2-inch diameter PiG and a unique process of wet-type CIP, we have successfully fabricated and demonstrated the good uniformity and excellent performances of a large diameter of 2-inch PiG. The proposed of a large diameter of 2-inch PiG with excellent performance, high reliability, and thermal stability is essential to meet the economic dimension requirement for commercial usages in high-quality WLEDs indoor lighting applications.

2. Experimental Details

2.1 Fabrication of a Diameter of 13-mm Phosphor-in-Glass

The glass matrix of B_2O_3-Sb_2O_3-SiO_2-Ta_2O_5 were prepared to fabricate the PiG: B_2O_3, Sb_2O_3, SiO_2, and Ta_2O_5. The mixed raw materials were heated in a platinum crucible at 1080 °C for 1 hour to melt. After cooling, the glass matrix (B_2O_3-Sb_2O_3-SiO_2-Ta_2O_5) was milled into glass powders and the particle size of about 10 μm was selected. Fluorescence spectrometer (HITACHI F-4500) was used to measure the photoluminescence excitation (PLE) spectra of the red and green PiG to select the appropriate phosphors. $CaAlSiN_3$: Eu^{2+} (red) and $Lu_3Al_5O_{12}$: Ce^{3+} (green) phosphors were selected as red and green luminescence centers, respectively, and the particle size of these

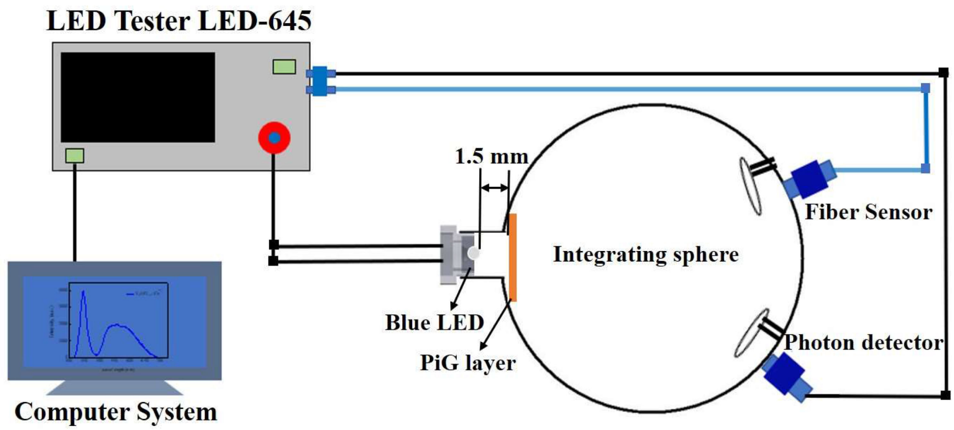

Fig. 1. The schematic structure of the remote PiG-based WLED.

TABLE 1
Five Manufacture Conditions of the PiG

Formula	G5:R1	G6:R1	G8:R1	G9:R1	G5:R1
Total amount of mixture (g)	10	10	10	10	100
Amount of glass matrix (g)	5	6.16	7.4	8	50
Amount of $Lu_3Al_5O_{12}$: Ce^{3+} (g)	4.167	3.291	2.311	1.8	41.67
Amount of $CaAlSiN_3$: Eu^{2+} (g)	0.833	0.549	0.289	0.2	8.33
Sintering temperature (°C)			620		620
Diameter			13-mm		2-inch
Thickness (mm)			0.18/0.20/0.22/0.24/0.26		0.18

two phosphors was about 15 μm. These two phosphors showed the wide spectral width of full width at half maximum (FWHM) broad emission spectra and the appropriate emission spectra peaks. Finally, red, and green phosphors with various proportions and weight ratios were uniformly blended into the glass matrix, and the uniform mixture powder was pressed uniaxially to form a formed precursor of PiG, and then sintered into a block at a low temperature of 620 °C for 30 minutes, followed by cooling to room temperature. The resulting PiG with a diameter of 13 mm was cut into the thicknesses of 0.18-, 0.20-, 0.22-, 0.24-, and 0.26-mm. Use an X-ray diffraction analyzer (XRD, BRUKER D8 SSS) to evaluate the typical X-ray diffraction powder patterns to confirm whether there was any chemical change during the sintering process. The schematic structure of the remote PiG-based WLED was shown in Fig. 1. The blue LED as a pumping source was installed on a temperature-controllable heat sink, and the temperature of the heat sink was controlled at 25 °C during the measurement. The driving current of the pumping source was 1000 mA, which was provided by the LED Tester LED-645 (Isuzu OPTICS ISM-360 series). The light emission spectra, CIE 1931 (x, y) chromaticity coordinates, CCT, CRI, and luminous efficiency of the PiG layer were carried out by using the GaN-based 450 nm blue LED as the pumping source, an integrate sphere equipped with an optical fiber, and a CCD detector as the spectra recorder. Through experimental analyses, the PiG with high CRI in a complete range of white light CCTs were realized by four manufacture conditions of the G5:R1, G6:R1, G8:R1, and G9:R1, as listed in Table 1. The weight ratios of all phosphors and proportions of green and red phosphors for the G5:R1, G6:R1, G8:R1, and G9:R1 were 50wt% ($4.167+0.833 = 5$) and 5:1 (4.167:0.833), 38.4wt% ($3.291+0.549 = 3.84$) and 6:1 (3.291:0.549), 26wt% ($2.311+0.289 = 2.6$) and 8:1 (2.311:0.289), and 20wt% ($1.8+0.2 = 2$) and 9:1 (1.8:0.2), respectively.

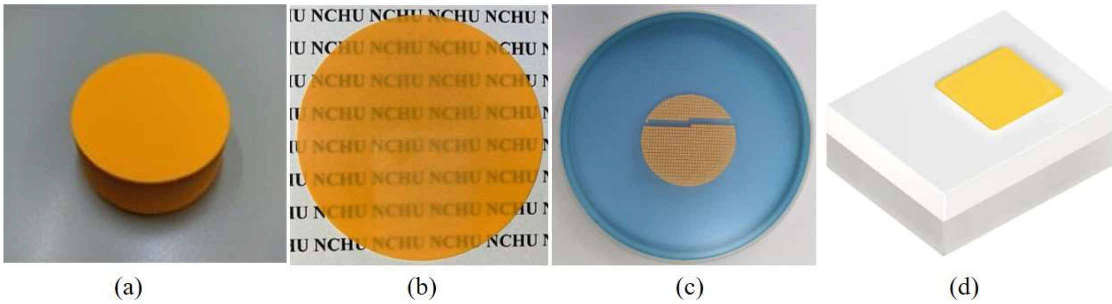

Fig. 2. The related photos of a diameter of 2-inch PiG.

2.2 Fabrication of a Diameter of 2-Inch Phosphor-in-Glass for WLEDs Package

As shown in Table 1, the best formula of G5:R1 was as following to produce a diameter of 2-inch PiG, the total amount of the mixture powder was 100 g and the weight ratio of the glass matrix, green phosphor, and red phosphor in the mixture powder were 50wt%, 41.67wt%, and 8.33wt%, respectively. In this work, a novel wet-type cold isostatic pressing (CIP) technique was performed to achieve a diameter of 2-inch PiG with excellent optical performance, high reliability, and thermal stability. The uniform mixture powder was pressed uniaxially to form a formed precursor, then the formed precursor was covered with a plastic rubber mold, and placed in a cavity filled with medium liquid, and finally processed by wet-type CIP at 250 MPa for 15 minutes, to remove the trapped pores and form a formed precursor with a high uniform bulk density, to be sintered at a low temperature of 620 °C for 30 minutes, and then cooled to room temperature. According to the specifications of the final product, the resulting PiG with a diameter of 2-inch was cut into the PiG with appropriate thickness, and then the double-sided surface of the PiG was polished, and the total thickness variation of the PiG was controlled within 5 μm to obtain the PiG with uniform optical characteristics. FE-SEM (JEOL JSM-7800F) was used to determine the surface microstructure of PiG with and without wet-type CIP process to confirm the effectiveness of the wet-type CIP process, and used an integrating sphere measurement system to evaluate the uniformity of optical performances. Figs. 2(a), 2(b), 2(c), and 2(d) show a diameter of 2-inch PiG bulk, a diameter of 2-inch PiG with a thickness of 0.18 mm, a dimensions of $1.25 \times 1.6 \times 0.18$ mm PiGs on a diameter of 6-inch blue tape for automatic placement machine, and a WLED package with a dimensions of $1.25 \times 1.6 \times 0.18$ mm PiG for reliability tests, respectively.

3. Results and Discussion

Fig. 3 shows the normalized PLE spectra and light emission spectra of the red and green PiG. The green PiG showed a peak at 518 nm with broad emission of FWHM 120 nm and the red PiG indicated a peak at 650 nm with broad emission of FWHM 90 nm. The PLE spectra of the red PiG overlapped with the light emission spectra of the green PiG and part of the green light emission was reabsorbed by the red phosphor, thereby increasing the red emission. The reabsorption in multiple phosphor systems may lead to energy loss and the luminous efficiency reduction of WLEDs. Fig. 4 shows the typical X-ray diffraction powder patterns of the red and green phosphors and PiG. The pattern indicated that the two various phosphors coexisted in the PiG. The pattern also showed the presence of the red and green phosphors in the PiG, which indicated that the chemical properties of the red and green phosphors did not change during the sintering process.

3.1 Measurement and Discussion of a Diameter of 13-mm PiG

In this work, the precisely fabricated processes are used to produce the various golden formulas of the diameter of 13-mm PiG with high CRI over 90 in a complete range of white light CCTs.

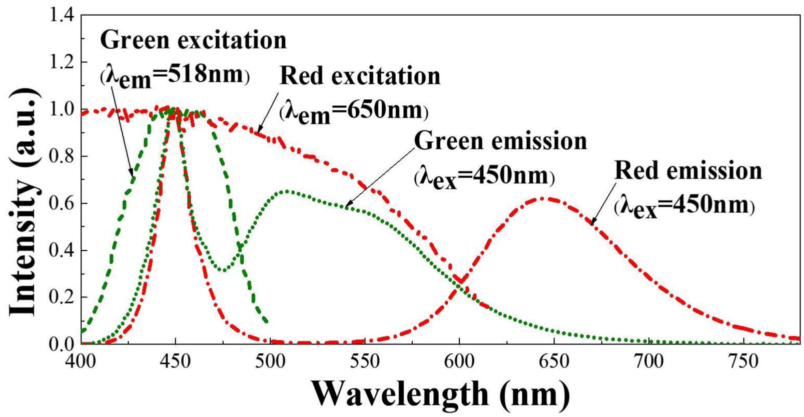

Fig. 3. Photoluminescence excitation and light emission spectra of the red and green PiG.

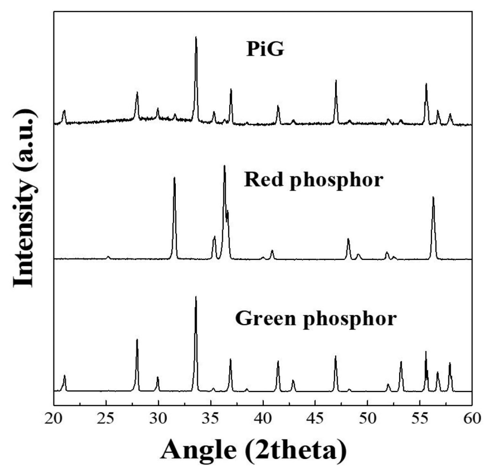

Fig. 4. Typical X-ray diffraction powder patterns of the red and green phosphors and PiG.

Through experimental analyses, the PiG with high CRI in a complete range of white light CCTs were realized by using four various golden formulas based on only one combination of $CaAlSiN_3$: Eu^{2+} (red phosphor) and $Lu_3Al_5O_{12}$: Ce^{3+} (green phosphor), as mentioned earlier in the Table 1. The measurement results of the G5:R1, G6:R1, G8:R1, and G9:R1 were shown in Figs. 5 and 6 and Tables 2, 3, and 4. The CIE 1931 (x, y) chromaticity coordinates of the WLEDs employing the four various golden formulas of PiG were shown in Fig. 5. The results showed that the WLEDs employing the PiG could achieve high CRI of over 90 in any CCTs range. The CCT is an important parameter of color appearance and is related to the nominally cold, or neutral, or warm state of the white light. WLEDs employing the PiG showed the complete range of white light CCTs from 2100 K to 7600 K, which included cold, neutral, and warm white light. A high CRI of over 90 could also be maintained over the complete range of white light CCTs, especially in the CCTs range from 2100 K to 3500 K, the CRI was greater than 97. The color temperature and emission spectra of pc-WLED can be tailored by mixing various phosphors of red, orange, yellow, and green emission wavelengths in various proportions and weight ratios. In this study, by using four various golden formulas based on only one combination of red and green phosphors, the complete range of white light CCTs from 2100 K to 7600 K and CRI greater than 97 were obtained. A complete range of white light CCTs was due to the precise adjustment of the thickness 0.18-0.26 mm to achieve the chromaticity tailorable PiG. In addition, the CRI greater than 97 was attributed by the optimized phosphor materials that had FWHM broad emission of 120 nm at the peak of 518 nm (green

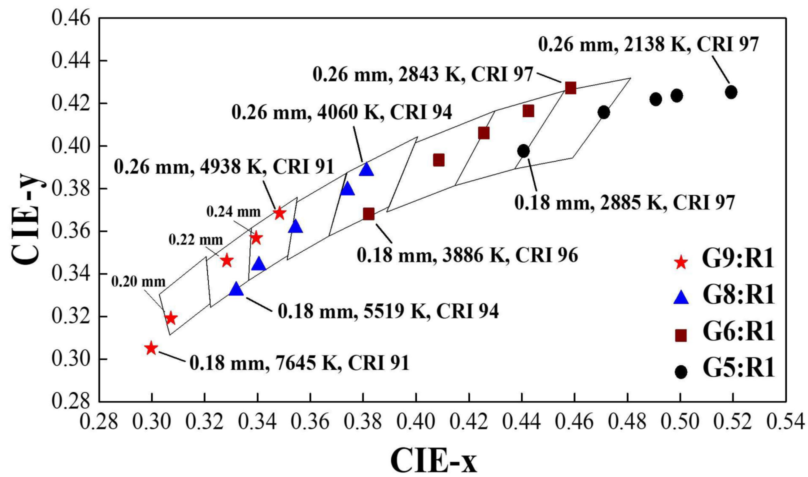

Fig. 5. CIE 1931 (x, y) chromaticity coordinates of the WLEDs employing the various formulas of the PiG.

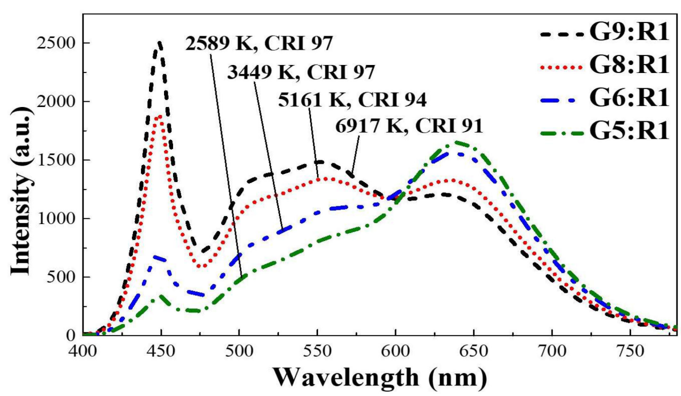

Fig. 6. Emission spectra of the WLEDs employing the various formulas of the 0.20 mm-thickness PiG.

TABLE 2
Performances of the WLEDs Employing the Various Formulas of the PiG At Various Thicknesses

Formula	Thickness (mm)	CIE (x, y)	CCT (K)	CRI (Ra)	Efficiency (lm/W)
G5:R1	0.22	(0.490, 0.421)	2398	97	46
G5:R1	0.20	(0.471, 0.415)	2589	97	48
G5:R1	0.18	(0.440, 0.397)	2885	97	51
G6:R1	0.22	(0.425, 0.406)	3215	97	57
G6:R1	0.20	(0.408, 0.393)	3449	97	58
G6:R1	0.18	(0.382, 0.368)	3886	96	60
G8:R1	0.24	(0.374, 0.379)	4195	94	66
G8:R1	0.22	(0.354, 0.362)	4702	94	67
G8:R1	0.20	(0.341, 0.344)	5161	94	68
G9:R1	0.22	(0.328, 0.346)	5676	91	72
G9:R1	0.20	(0.307, 0.319)	6917	91	72

TABLE 3
CRI Values for R1-R9 in Various Formulas of the PiG

Formula	Thickness (mm)	R1	R2	R3	R4	R5	R6	R7	R8
G5:R1	0.20	96	99	94	94	96	98	98	97
G6:R1	0.20	99	97	92	96	98	95	96	97
G8:R1	0.20	96	93	88	91	96	91	94	96
G9:R1	0.20	93	91	87	91	92	87	93	94

TABLE 4
R9-R15 Special CRI Values and Rf, Rg in Different Formulas of the PiG

Formula	Thickness (mm)	R9	R10	R11	R12	R13	R14	R15	Rf	Rg
G5:R1	0.20	97	98	91	98	97	95	98	96	103
G6:R1	0.20	99	92	95	86	98	95	99	95	102
G8:R1	0.20	98	85	91	80	95	93	98	92	102
G9:R1	0.20	84	78	91	71	92	93	94	85	100

phosphor) and 90 nm at the peak of 650 nm (red phosphor). The results clearly showed that the CCTs range could meet the CCTs value of the most commercial light sources (2700–6500 K) with CRI over 90 to apply the most high-quality indoor lighting markets.

The performances of the WLEDs employing the four various golden formulas of PiG with various thicknesses were summarized in Table 2. The CCTs range from 2398 K (warm colors) to 6917 K (cool colors), and the CRI exceeds 90. The CCT and CIE (x, y) of the WLEDs were precisely tailored by the various formulas and thickness of the PiG. The CCT decreased as both of excited blue LED light and the intensity of green color decreased, and intensity of red color increased. These were due to the amount of both red and green phosphors increased as the weight ratio increased. Similarly, in the same formula, the amount of both red and green phosphors increased as the thickness increased. The blue LED light as the pumping source decreased causing by the absorption of the red and green phosphors. The intensity of green color decreased as the amount of red phosphor increased. This was because of the reabsorption of the red phosphor. Therefore, the intensity of the red color at 650 nm increased as the amount of red phosphor increased. The luminous efficiency decreased as the weight ratio increased in various formulas. Similarly, in the same formula, the luminous efficiency decreased as the thickness increased. This was because the excitation spectra of the red phosphor overlapped with the emission spectra of the green phosphor, and part of the green light emission was reabsorbed by the red phosphor, thereby the luminous efficiency reduction of the WLEDs. The results of CCT and luminous efficiency were consistent with Fig. 6. Fig. 6 shows the emission spectra of the WLEDs employing the various formulas of the 0.20 mm-thickness PiG. The blue color intensity at 450 nm indicated that it decreased as the weight ratio of the phosphors increased, and the blue LED light as the pumping source was absorbed by the phosphors. The intensity of the light band with a center wavelength of 518 nm also decreased as the weight ratio increased. This was because of the reabsorption of the red phosphor, and therefore, as the weight ratio of the phosphors increased, the intensity of red color at 650 nm increased. The red phosphor reabsorbed the green emission light of the green phosphor, thereby the red emission light increased. This result could introduce of the lower CCT and luminous efficiency. Table 3 indicated that the CRI (Ra) was the average value of R1-R8 and the R1-R8 of G8:R1 and G9:R1 were significantly worse than G5:R1 and G6:R1. This was why the CRI (Ra) of G8:R1 and G9:R1 could not be the same as G5:R1 and G6:R1. Table 4 lists the special CRI values for R9-R15, which were not assessed when counting the common CRI (Ra) values. The light sources with high R9 (red) and R13 (skin tone) values, as well as high CRI (Ra), can make the skin color look healthier and natural. As shown in Table 4, R9 and R13 of these four formulas were

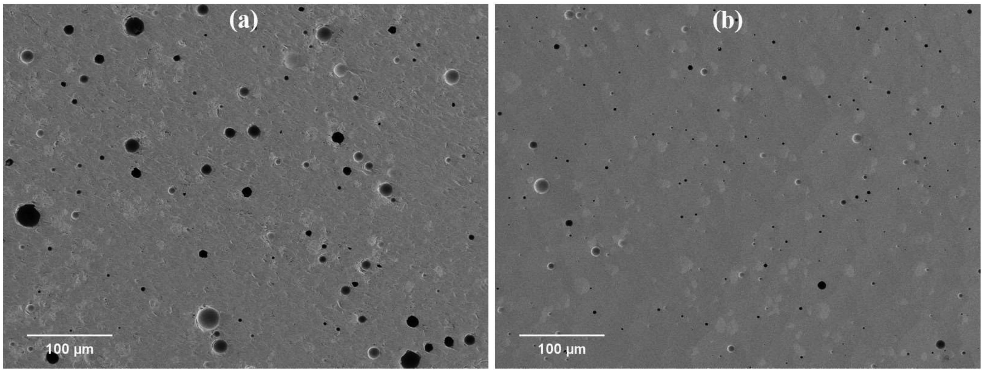

Fig. 7. FE-SEM micrographs of a PiG (a) without wet-type CIP process and (b) with wet-type CIP process.

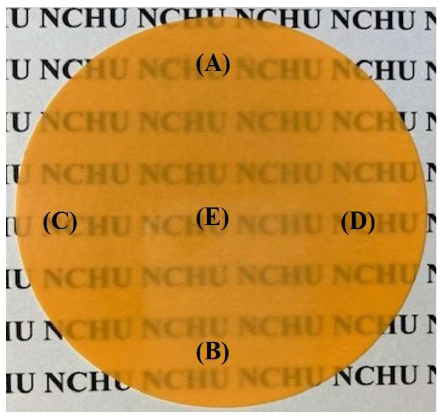

Fig. 8. A diameter of 2-inch PiG with a thickness of 0.18 mm.

TABLE 5
Five-Corner Measurement Results of a Diameter of 2-Inch PiG With a Thickness of 0.18 Mm

Measuring position	CIE (x, y)	CCT (K)	CRI (Ra)	Luminous Efficiency (lm/W)
Point (A)	(0.443, 0.402)	2882	97	51.12
Point (B)	(0.442, 0.403)	2905	97	51.21
Point (C)	(0.440, 0.399)	2908	97	51.22
Point (D)	(0.440, 0.402)	2931	97	51.27
Point (E)	(0.441, 0.398)	2886	97	51.13

in the range of 99-84 and 98-92, respectively. We further evaluated the color rendering, the new color index (IES TM-30-2015) was employed in this study, which consists of a fidelity index (Rf), and a gamut index (Rg) [14], [15]. The results were also shown in Table 4. The Rf and Rg of these four formulas were in the range of 96-85 and 103-100, respectively. As the results of CRI (Ra), R9, R13, Rf, and Rg, this indicates that the proposed PiG is excellent color rendering for high-quality WLEDs indoor lighting application.

3.2 Measurement and Discussion of a Diameter of 2-Inch PiG With a Thickness of 0.18 mm

An optimum approach for fabrication of a diameter of 2-inch PiG with a thickness of 0.18 mm was demonstrated. The results were shown in Figs. 7, 8 and Table 5. Fig. 7 shows the FE-SEM micrographs of a PiG (a) without wet-type CIP process and (b) with wet-type CIP process. Phosphor particles with a diameter of about 10–20 μm were distributed over the glass matrix. The porosity of a PiG (a) without wet-type CIP process and (b) with wet-type CIP process, estimated from the micrographs, were in the range of 2.93% and 0.37%, respectively. From the measurement result, the porosity of the PiG with wet-type CIP process was greatly reduced. As shown in Fig. 8, we further used an integrating sphere measurement system to determine the uniformity of the optical performances at points (A), (B), (C), (D) and (E). The results of optical performances were shown in Table 5. The differences in CIE (x, y) and CCT at points (A), (B), (C), (D) and (E) were less than 5 per thousand and 50 K, respectively, as shown in Table 5. The CRI (Ra) was the same at points (A), (B), (C), (D) and (E) and the average luminous efficiency was about 51.19 lm/W. The results showed that the precisely fabricating process achieved a diameter of 2-inch PiG with good uniformity and excellent performances by the five-corner measurement.

3.3 Reliability Tests of a Diameter of 2-Inch PiG With a Thickness of 0.18 mm

The reliability tests of a diameter of 2-inch PiG with a thickness of 0.18 mm were studied through the reflow, steady state operating life, and high temperature/humidity storage tests. In order to prepare the reliability samples, the blue LED chips were attached on a large ceramic substrate in order, and then a PiG with a dimensions of $1.25 \times 1.6 \times 0.18$ mm was covered on each blue LED chip, and then filled with white glue to block the blue light leaking from the gap, and finally diced into small dies, as shown in Fig. 2(d). The purpose of the reflow test was to confirm the thermal quenching effect of the PiG. The test temperature was 260 °C for 10 seconds, and then 3 times for each sample. The steady state operating life test was to perform an aging test of the PiG, which mainly verified the attenuation of the PiG caused by blue light excitation. The operating current of the blue LED was 1000 mA for 1008 hours testing time at room temperature. The high temperature/humidity storage test was to verify the attenuation of the red phosphor. The test temperature and humidity were 60 °C and 90%, respectively, for 300 hours testing time.

Before and after the reliability tests, the optical properties of the WLEDs including luminous efficiency, CRI, and CIE (x, y) were measured to evaluate the thermal stability and reliability of the PiG. The reliability test pass/fail criterion was that the change before and after the reliability test was less than 1dB. The number of samples for each test item were 11pcs. The results of the reliability tests for the PiG showed that the changed in the optical properties of CIE (x, y), CRI, and luminous efficiency were all less than 1 dB. This indicates that the proposed PiG is excellent reliability and thermal stability.

4. Conclusion

In summary, by using four various golden formulas based on only one combination of $CaAlSiN_3$: Eu^{2+} (red phosphor) and $Lu_3Al_5O_{12}$: Ce^{3+} (green phosphor), the complete range of white light CCTs from 2100 K to 7600 K and CRI greater than 97 in the PiG-based WLEDs were obtained. A complete range of white light CCTs was due to the precise adjustment of the thickness 0.18–0.26 mm to achieve the chromaticity tailorable PiG. In addition, the CRI greater than 97 was attributed by the optimized phosphor materials that had FWHM broad emission of 120 nm at the peak of 518 nm (green phosphor) and 90 nm at the peak of 650 nm (red phosphor). Based on the best formula of a 2-inch diameter PiG and a unique process of wet-type CIP, we had successfully fabricated and demonstrated good uniformity, excellent performances, and high reliability for a large diameter of 2-inch PiG. The results of the 2-inch diameter PiG may be directly placed on a blue tape with a diameter of 6-inch, which is used in automatic placement machine for automated production. The proposed of a large diameter of 2-inch PiG with excellent performance, high reliability, and thermal stability is essential to meet the economic dimension requirements for commercial usages in high-quality WLEDs indoor lighting applications.

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