Facile Synthesis of BCNO Yellow-Emitting Phosphors Applications for White LEDs by Microwave Heating

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Abstract Yellow-emitting BCNO phosphors, applied for white light-emitting-diodes (LEDs), were synthesized by a facile microwave heating route at lower temperature within short duration. The prepared BCNO phosphors exhibited amorphous form and tunable yellow emission in the range of 510–550 nm under the excitation of 450-nm blue light. The effects of carbon content on the photoluminescence properties for these BCNO phosphors and their application performances in white LEDs were investigated in detail. The demonstrated microwave synthesis route is promising in preparing low-cost phosphors, and the prepared BCNO phosphor may find potential application in blue-based white LEDs.

KEY WORDS: BCNO phosphors; Photoluminescence; Microwave heating; White LEDs

1 Introduction

White light-emitting-diodes (LEDs), an alternative to traditional incandescent bulbs and fluorescent lamps, have received considerable attention in the past decades owing to its merits in long lifetime, low power consumption, high efficiency, and reliability [1–3]. The performances of phosphor-converted white LEDs, such as luminous efficiency, color rendering index (CRI), and correlated color temperature (CCT), are strongly dependent on the photoluminescent properties of phosphors. The discovery and

development of phosphors applied in WLEDs have moved from a single family of phosphor compound, the Ce³⁺ doped Y₃Al₅O₁₂, to a plethora of rare-earth (RE) ions doped silicates, sulfides, nitrides, and molybdates [4–6]. These RE ions play a crucial role in controlling the luminescence of these phosphors. However, most of the RE resources are suffering from the exhaustion because of the rare feature, which results in the expensive production cost for rare earth containing phosphors. Thus, the exploitation of rare-earth-free phosphors has been a long-standing and significant issue in the coming development of phosphors.

The BCNO phosphor, a good candidate as one of the rare-earth free phosphors, was firstly exploited by Ogi et al. [7]. And then, an abundant of efforts had been focused on the synthesis and luminescent properties of BCNO phosphors [8–15]. As the name implies that BCNO phosphors are essentially composed of boron (B), carbon (C), nitrogen (N), and oxygen (O) atomic compositions. Heat condensation method, sintering at 600–900 °C for several hours, was widely used to prepare BCNO phosphor from aqueous solutions or powders of boron, carbon, and nitrogen sources. Ogi et al. [7] synthesized BCNO phosphors by heating boric acid, urea, and PEG precursors mixture at 700–900 °C for

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30–60 min. Zhang *et al.* [16] prepared BCNO phosphors by sintering boric acid and hexamethylenetetramine raw materials at 600–700 °C for 4–24 h. To produce BCNO nanoparticles, Lei *et al.* [10] synthesized BCNO nanophosphor in a eutectic LiCl/KCl salt melt at 750–850 °C for 1–2 h by using sodium borohydride and urea as raw materials. Very recently, 2D BCNO nanophosphor was prepared by heating boric acid, urea or carbamide, and PED at 750–800 °C about 1 h [14]. Nevertheless, high-temperature and long-duration heating process in an electric furnace for heat condensation method is high-energy consumption. For taking lower production cost of BCNO phosphors into consideration, it is desirable to prepare BCNO phosphors with a lower temperature and shorter reaction time. Microwave heating, as a fascinating “green chemistry” processes, is applied to synthesize a wide variety of organic and inorganic materials [17, 18]. Microwave heating has the following advantages, over the conventional heating, uniform heating, rapid heating, and high effective heating. As a result, microwave heating requires much less energy than conventional methods. Much lower temperature and shorter reaction times are prominent for microwave heating than the electric furnace heating in synthesis of BCNO phosphors. To the best of our knowledge, however, there are only a few works pertaining to the microwave synthesis of BCNO phosphors demonstrated by Ogi’s group [19, 20]. For the luminescent properties, reported works had evidenced that the emission properties for BCNO phosphors are strongly dependent on the synthesize conditions, such as raw materials, sintering temperature, sintering time, and ambient atmosphere. Ogi *et al.* [7] investigated the effects of sintering time and temperature and ratio of raw materials on luminescent properties of BCNO phosphors and found that the emission spectra were tuned from 387 to 570 nm ($\lambda_{\text{ex}} = 365$ nm) with increasing ratio of C/B. Zhang *et al.* [13] disclosed the obviously redshift of emission wavelength in the range from 450 nm to 570 nm ($\lambda_{\text{ex}} = 370$ nm) for blue-emitting BCNO phosphors, resulting from the change in sintering temperature and the ratio of raw materials. The same group also reported the scarce red emission in BCNO phosphors [16]. It is well known that the presently commercial white LEDs are encapsulated by the combination of a blue LED chip and a yellow phosphor. Phosphors applied in blue-chip-based white LEDs should substantially meet two basic requirements. One is the emitting light of phosphors locates in the yellow region so as to realize white light with combination of blue LED chip. The other is the optimal excitation wavelength of phosphors matches well with the emitting light of blue LED chip. Regrettably, BCNO phosphors reported in above-mentioned precedents are

seldom suitable to be applied in blue-chip-pumped white LEDs, because of the improper light emission of phosphors in non-yellow region or the mismatch between excitation light of phosphors and emit light of blue LED chip.

In this study, yellow-emitting BCNO phosphors application for blue-chip-pumped white LEDs was synthesized by a facile microwave heating route in a household microwave oven at lower temperature with shorter reaction times. The effects of carbon content on the photoluminescence properties for the prepared BCNO phosphors and the application performances of phosphor in blue-based white LEDs were investigated in detail. The demonstrated microwave synthesis route is promising in preparing low-cost BCNO phosphors, and the prepared yellow-emitting BCNO phosphor may find potential application in blue-based white LEDs.

2 Experimental

The BCNO phosphors were prepared by the precursor: boron acid as the boron source, citric acid as the carbon source, and urea as the nitric source. All the reagents were purchased from Aladdin Industrial Corporation and used without purification. The molar ratio of the nitride/boron (N/B) was fixed at 2.0, and the molar ratio of the carbon/boron (C/B) was varied from 2.5 to 20.0. Precursor solution was prepared by dissolving 0.31 g boron source, 0.30 g urea, and suitable amount of citric acid (0.44, 0.88, 1.76, 2.64, and 3.52 g) in 20-mL ion-exchanged water and stirred for 30 min at room temperature. Then, the solution was placed in a 600-W household microwave oven and heated by microwave radiation for 15 min to water exsiccation and then for 1 min to obtain the BCNO phosphors. A white LED prototype was fabricated with a commercial 460-nm blue chip and our prepared BCNO phosphor.

The crystalline structure of the prepared BCNO phosphors was analyzed by X-ray diffractometer (XRD, Rigaku, RINT Ultima-III, Japan) with $\text{CuK}\alpha$ radiation. The particle morphology and the elemental analysis were measured using a scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS) (Hitachi, S-4800). X-ray photoelectron spectroscopy (XPS, PHI1600EXCA) was used to characterize the chemical state of BCNO phosphors. The chemical bonding status was measured by Fourier transform infrared spectroscopy (FTIR, Bruker, WQE-410). The ultraviolet–visible (UV–Vis) absorption spectra were measured by a UV–visible spectrometer (TU-1901, China) with a 60-mm-diameter integration sphere. Photoluminescence (PL) and photoluminescence excitation (PLE) spectra as well as the absolute quantum efficiency (QE) for samples were

measured on a fluorescence spectrometer (Hitachi F-4600, Japan) equipped with an integrating sphere (Orien KOJI, QY-2000, China). Commission International de l'Eclairage (CIE) chromaticity performances for the phosphors and the fabricated LED prototypes were traced on PMS-80 Plus UV–Vis–near-IR Spectro-photocolorimeter (EVERINE, China) system equipped with a phosphor excitation equipment ($\lambda_{\text{ex}} = 460 \text{ nm}$) and an integrating sphere.

3 Results and Discussion

Figure 1 shows the XRD patterns of the prepared BCNO phosphors with variable C/B molar ratios. A broad bump centered at around 20° in XRD pattern was recorded for all samples, indicating that the as-prepared BCNO phosphors by microwave heating is non-crystalline. This observation is obviously different from the results obtained by traditional furnace heating. The presence of crystalline B_2O_3 , h-BN, and carbon was indexed through XRD diffraction peaks in BCNO phosphor by Nuryadin *et al.* [21]. Zhang *et al.* [16] disclosed that their prepared BCNO phosphor showed turbostratic boron nitride (t-BN) structure accompanied with cubic B_2O_3 . In another Zhang's work, XRD results suggested that the prepared BCNO phosphor was composed of mainly cubic B_2O_3 and partially crystalline BCNO, while the crystalline h-BN had not been formed [22]. Apparently, the crystalline BN indexed on BCNO phosphor is thought to originate from the exothermic reaction of boron and nitrogen sources, while crystalline B_2O_3 is produced by the thermal decomposition of unreacted boron sources during the higher temperature and longer duration of heating. For the microwave heating prepared BCNO phosphor, non-crystalline production is

ascribed to the lack of enough energy to the crystal growth of BN and B_2O_3 at a lower temperature and shorter duration of microwave heating comparing with the conventional heating. Similar non-crystalline BCNO phosphors were prepared by Ogi *et al.* through microwave heating [19]. However, crystalline BCNO phosphor was reported in their other work, in which microwave-assisted heat was inserted into the traditional furnace heating procedure [20].

To investigate the chemical state of the prepared BCNO phosphor, FTIR analysis in the range of $400\text{--}4000 \text{ cm}^{-1}$ was performed as shown in Fig. 2. The broadband at 3250 cm^{-1} is attributed to hydroxyl group, indicating that the entire hydroxyl group did not derive from the citric acid undergoes dehydration condensation [19]. The absorption at ~ 765 and $\sim 1387 \text{ cm}^{-1}$ is related to the B–N–B vibration and B–N stretching mode, which verifies the formation of B–N linkage in the non-crystalline BCNO phosphor [22]. The B–C and C–N vibration peaks are observed at ~ 1197 and $\sim 1247 \text{ cm}^{-1}$, respectively [10, 23]. The vibrations at ~ 645 and $\sim 1430 \text{ cm}^{-1}$ correspond to B–O bond, and the vibration at 885 cm^{-1} corresponds to stretching vibration of tetrahedral BO_4^- units in B_2O_3 [22]. The N–B–O stretching band in the $900\text{--}1100 \text{ cm}^{-1}$ region is clearly shown. The broad peak in the neighborhood of 1720 cm^{-1} is induced by the vibration of C=O bond, indicating the presence of COOH, CONH, and other carbonyl compounds. From the FTIR analysis, it can be concluded that the non-crystalline BCNO phosphor is obtained by the bonding of B, C, N, O elements to each other.

Figure 3 shows the elemental composition of the non-crystalline BCNO phosphor with C/B of 5.0 as determined by EDS analysis. The result indicates that the obtained sample is composed of boron, carbon, nitride, and oxygen,

Fig. 1 XRD patterns for the prepared BCNO phosphor with C/B molar ratio 5.0

Fig. 2 FTIR of the prepared BCNO phosphor with C/B molar ratio 5.0

Fig. 3 EDS elemental analysis and SEM image for the prepared BCNO phosphor with C/B molar ratio 5.0

which constitute a BCNO phosphor. The atomic percentage for B, C, N, and O is measured to be about 21.40%, 36.35%, 13.20%, and 29.05%, respectively. The evaluated C/B molar ratio is much lower than the nominal value of 5.0. The SEM image (inset of Fig. 3) displays that the prepared BCNO phosphors is irregular in shape, and the particle size was in the range of 1–3 μm . In Ogi's work, nanosized BCNO particles with approximation of 50 nm in size were obtained by the microwave heating [19]. They proposed that the short reaction time of 120 s under microwave heating shows contribution to the nanosized production. For our samples, the longer duration of microwave heating should be responsible for the larger-size production.

To further investigate the chemical structure of the as-obtained BCNO phosphor, XPS spectra is measured as shown in Fig. 4a–e. Several distinct peaks around 190, 283, 400, and 534 eV corresponding to the B 1s, C 1s, N 1s, and O 1s peak (Fig. 4a) are observed, respectively [16, 22]. From the high resolution of XPS analysis, the high-resolution B 1s spectrum shown in Fig. 4b can be fitted by Gaussian curves with dominating components centered at 190.4 and 191.0 eV. The binding energy peak at 190.4 eV is ascribed to B–O or B_2O_3 bond, while the peak at higher energy is attributed to B–N. The C 1s peak (Fig. 4c) can similarly be deconvoluted into three peaks centered at 282.9, 284.6, and 286.9 eV, corresponding to C–C, C–O, and O–C=O bond, respectively. The N 1s peak (Fig. 4d) shows two peaks at 397.9 and 399.8 eV, which is attributed to the signature of B–N and C–N bonds, respectively. Regarding O 1s peak shown in Fig. 4e, two components were found centered at 530.3 and 531.8 eV, which may be attributed to the O=C and O–B bonds, respectively. From the quantitative analysis, the atomic percentage of the sample surface contains 30.55% carbon, 41.53% oxygen, 18.35% nitrogen, and 9.57% boron. The evaluated C/B

molar ratio from XPS analysis is much closer to the nominal value comparing with EDX analysis. Above observed XRD, FTIR, EDS, and XPS results imply that the obtained BCNO phosphor is non-crystalline constituted by many carbon and oxygen elements with a small quantity of boron and nitrogen.

Figure 5 shows the normalized PL and PLE spectra for the prepared BCNO phosphor with C/B molar ratio of 5.0. Broad yellow-green emission band with maximum wavelength at 525 nm is observed on the PL spectrum under the excitation of 450 nm. The PLE spectrum monitored at 525 nm for the prepared BCNO phosphor is recorded in the wavelength range of 300–500 nm, indicating the potential application in blue-chip-pumped white LEDs. The full width at half maximum for this yellow emission BCNO phosphor is evaluated to be about 100 nm, which allows for good color rendering of illuminations for white LEDs. The observed photoluminescent properties for as-prepared non-crystalline BCNO phosphor are very likely to the reports in Ogi's work [19]. Unlike other phosphor materials, in which the luminescence is generated by the active rare-earth ions, such as Ce^{3+} , Eu^{2+} , and Dy^{3+} , the emission of BCNO phosphor is induced by the closed-shell BO_2^- and BO^- species or the defects of energy-level transition [11, 16]. About the luminescent mechanism for BCNO phosphor, the previously explanation is based on the understanding that BCNO is crystalline intermediates between graphite and hexagonal-BN [7]. At present, there are mainly two points of view to clarify the luminescence origin of BCNO phosphors [16]. One argument is that the closed-shell BO_2^- and BO^- species are responsible for the luminescence of BCNO phosphor. Another suggestion is that the defect of energy-level transition results in the luminescence of BCNO phosphor. In view of the absence of crystalline B_2O_3 , h-BN, or carbon for our prepared amorphous BCNO phosphors, the probable luminescence origins of these samples are attributed to the closed-shell BO_2^- and BO^- species acting as luminescent centers or impurity defects in materials. Similar luminescent mechanism was proposed by Ogi et al. [19] for amorphous BCNO phosphor synthesized by microwave heating.

References had evidenced that the emission properties of BCNO phosphors are strongly dependent on the synthesis conditions, such as raw materials, sintering temperature, sintering time, and ambient atmosphere. At present, the main method to modulate the emission spectra is to select proper carbon source and to increase the concentration of carbon source. The influence of the carbon content on the photoluminescent properties for our microwave heating synthesized BCNO phosphors was investigated. Figure 6 depicts the normalized PL and PLE spectra for the microwave heating synthesized BCNO phosphors with variable C/B molar ratios. With the

Fig. 4 **a** XPS analysis for the prepared BCNO phosphor with C/B molar ratio 5.0, **b–e** deconvoluted patterns of B 1s, C 1s, N 1s and O 1s peak

Fig. 5 PL and PLE spectra for prepared BCNO phosphor with C/B molar ratio 5.0

Fig. 6 Normalized PL and PLE spectra for prepared BCNO phosphors with variable C/B molar ratios

increase in the carbon content, the emission peak shows an obvious redshift from 510 nm for $C/B = 2.5$ to 550 nm for $C/B = 10.0$ firstly. With further increasing carbon content, the emission peak then takes a blueshift to 535 nm ($C/B = 20.0$). Similarly, the emission peak position of BCNO shifted to long wavelength firstly and then to short wavelength with increasing carbon content (PEG mass), which was reported by Zhang *et al.* [24]. Their first-principle calculations showed that the spectra properties of BCNO phosphors can be affected by the energy levels in the band gap produced by the carbon and oxygen impurities. The observed redshift of emission

peak is induced by the composition of BCNO and especially the carbon impurity concentration, and the blue shift of emission peak is induced by the superfluous carbon impurity. Liu *et al.* [25] also disclosed that the changeable emission band of BCNO phosphor was found to be correlated with the concentration of the intentionally introduced carbon impurity, which has a stabilization effect in the presence of the paramagnetic center V_N . Thus, the observed changes in emission spectra for our samples are likely to be caused by the difference in the concentration of carbon impurities [16, 24, 25]. Upon increasing the impurity concentration, the impurity levels

would shift to a lower energy, resulting in the redshift of emission spectra. For the $C/B > 10.0$, the excess of carbon impurity results in the formation of carbon residue on the production, which might induce the blueshift. For the PLE spectra, similar redshift is recorded owing to the related change in carbon-related impurity levels' position.

Figure 7 exhibits the CIE diagram for the prepared BCNO phosphors with variable C/B molar ratios under the excitation of 450-nm blue light. CIE (x , y) coordinates for the lower carbon content samples ($C/B = 2.5$ and 5.0) are located at in greenish-yellow region, while the higher carbon content samples ($C/B = 10.0$, 15.0 , and 20.0) are recorded as typical yellow-emitting phosphors. With the increase in carbon content, the emitting color for the obtained BCNO phosphors became a yellow emitter under the excitation of blue light, implying the potential application in blue-chip-based white LEDs.

Luminescence quantum efficiency (QE) is very important for phosphor materials in the practical application. The absolute η_{QE} for the obtained BCNO phosphors is calculated by the following equation:

$$\eta_{QE} = L_S / (E_R - E_S),$$

where L_S is the emission spectrum of the studied phosphor sample, and E_S and E_R are the excitation profiles of the exciting light with the sample and without the sample in the sphere, respectively. The measured η_{QE} value of BCNO phosphors is determined as 3.9% ($C/B = 2.5$), 30.3% ($C/B = 5.0$), 2.7% ($C/B = 10.0$), 3.3% ($C/B = 15.0$), and 6.3% ($C/B = 20.0$), respectively. It is found that the η_{QE} increases with the increase in C/B molar ratio at firstly and then gets maximum value when C/B molar ratio is 5.0. As the C/B molar ratio exceeds 5.0, the η_{QE} values become lower. This change is strongly related to the carbon impurities, which is proposed to be the luminescence origin of BCNO phosphors. When C/B molar ratio is 5.0, suitable carbon impurity concentration occurs in the materials for a high luminescence. As C/B is larger than 5.0, the excess of carbon impurity results in the formation of carbon residue in phosphor, resulting in the dramatically decrease of η_{QE} values.

To demonstrate the application of the BCNO phosphors, a white LED prototype is fabricated by combining commercial 450-nm blue chip and prepared BCNO phosphor with C/B molar ratio 20.0. Figure 8 presents the electroluminescent spectrum of the white LED prototype driven by a forward bias current of 25 mA. On the spectrum, a sharp emission peak and a broad emission band are observed. Apparently, these two emissions originate from the 450-nm LED chip and the BCNO phosphor, respectively. It is found that the emission peak of BCNO phosphor in the LED prototype is detected to be around 600 nm, which is longer than the recorded emission peak (550 nm) in PL spectrum of BCNO phosphor. The reason for this peak difference is most probable ascribed to the sensitivity difference of the photoelectric detector in the red light region for the used photometers. The CIE chromaticity coordinates, correlated color temperature (CCT), and color rendering index (CRI) for this white LED prototype are measured to be (0.38, 0.35), 3600 K, and 86.7, respectively, indicating the potential application for the prepared BCNO yellow phosphors in

Fig. 7 CIE chromaticity coordinates for prepared BCNO phosphors with variable C/B molar ratios

Fig. 8 Electroluminescent spectrum of a white LED prototype encapsulated with a 450-nm blue chip and as-prepared BCNO phosphor. The inset is the photograph of the white LED driven by 25 mA forward bias current

white LEDs. Additionally, the performances of white LEDs can be easily tuned by tailoring the usage of BCNO phosphor.

4 Conclusion

In summary, yellow-emitting BCNO phosphors application for blue-chip-pumped white LEDs has been synthesized successfully by a facile microwave heating route in a household microwave oven at lower temperature with shorter reaction times. XRD, FTIR, EDS, and XPS revealed that the obtained BCNO phosphor is non-crystalline and composed of many carbon and oxygen elements with a small quantity of boron and nitrogen. The emission spectra of the BCNO phosphor could be easily tuned from 510 to 550 nm by controlling the carbon content. Excellent white emission with CIE chromaticity coordinates at (0.38, 0.35), CCT of 3600 K, and CRI of 86.7 is obtained by combining blue LED chip and the prepared BCNO phosphor. These results indicate that the proposed microwave synthesis route is promising in preparing low-cost BCNO phosphors, and the prepared yellow-emitting BCNO phosphor may find potential application in blue-based white LEDs.

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