Influence of Surface Passivation on Perovskite CsPbBr_{1.2}I_{1.8} Quantum Dots and Application of High Purity Red Light-Emitting Diodes

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Abstract

In this work, trioctylphosphine oxide (TOPO) ligand is employed to improve the quality of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ quantum dots (QDs) films. Lead nitrate ($\text{Pb}(\text{NO}_3)_2$) is also used to passivate the surface of the films. The study of ligand and surface passivation on the luminous efficiency of red light-emitting diode (LED) is discussed. The $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs films co-doped with TOPO and $\text{Pb}(\text{NO}_3)_2$ can effectively improve the performance of the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs LEDs due to reduction of non-radiation recombination of the carriers and smooth morphology in the active layer, thus improving the injection and transportation capabilities of carriers. As a result, the highest luminosity and current efficiency are 502.7 cd/m^2 and 0.175 cd/A , respectively.

1. Introduction

Perovskite is a mineral composed of calcium, titanium and oxygen, and its chemical formula is CaTiO_3 [1]. Any compound represented by the general formula ABX_3 can be called perovskite. Material of A site can be divided into all-inorganic perovskite and organic-inorganic mixed type perovskite. A is a monovalent inorganic cation. It is composed of Cs^+ . A can also be composed of monovalent organic cations, such as CH_3NH_3^+ (methylammonium, MA^+) or $\text{CH}(\text{NH}_2)_2^+$ (formamidinium, FA^+), B is composed of divalent metal cations, usually lead ion Pb^{2+} or tin ion Sn^{2+} , and X is a halogen anion, mainly Cl^- , Br^- or I^- [2-5]. By controlling the combination and ratio of A, B, and X, different optical properties can be adjusted. MAPbX_3 and CsPbX_3 active layer in device structure mainly used in solar cells, photodetectors, optoelectronic devices, and light-emitting diodes (LEDs) [6-14].

Surface passivation is an effective method to protect the surface of the perovskite film, reduce the charge traps caused by structural defects, and prevent the surface from being affected by moisture in the air, and improve its luminous efficacy. Several articles have reported the effects of surface passivation on the optical and electrical properties of materials, and demonstrated enhanced luminescence properties by inhibiting non-radiative recombination and reducing defect states [15-18]. Polyhedral siloxane oligomers (POSS) serve as a useful passivation layer. POSS-coated perovskite quantum dot light-emitting diodes improve water resistance or poor solvent resistivity and film-forming ability. The brightness and stability of light-emitting diodes treated with POSS are improved [19]. The tri-n-octyl phosphine oxide (TOPO) is passivated on the surface of the perovskite FAPbBr_3 film to reduce the non-radiative recombination on the surface of the perovskite or the grain boundary [20]. After using AX ($\text{A}=\text{FA}^+$, MA^+ or Cs^+ , and $\text{X} = \text{Br}^-$ or I^-) to treat the surface of the CsPbI_3 perovskite film, it greatly improves the electronic coupling between quantum dots, thereby enhancing carrier transportation rate [21]. This work studies the characteristics of all-inorganic perovskite $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs with TOPO and $\text{Pb}(\text{NO}_3)_2$ surface passivation treatment. And, the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs films with TOPO and $\text{Pb}(\text{NO}_3)_2$ were used as the active layer to examine the optoelectronic properties of red light-emitting diodes (LEDs).

2. Experiments

2.1 Preparation of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$:TOPO solution

First of all, 0.132 M cesium carbonate (Cs_2CO_3), 20 mL octadecene (ODE), and 1.25 mL oleic acid (OA) were added into a 50 mL beaker A, and heated it at 120°C for 1 hr. The temperature was then increased to 150°C for 15 min to make the cesium carbonate and oleic acid react completely, and the solution appeared light yellow color. Next, TOPO with different density (1.0, 0.2, and 0.4 mmol), 0.3 mmol of lead bromide (PbBr_2), 0.45 mmol of lead iodide (PbI_2), 20 mL of octadecene (ODE), 2 mL of oleic acid (OA), and 2 mL of oleylamine (OAm) were added into a 50 mL beaker B, and heat it at 120°C for 1 hr. Then, the temperature raised up to 180°C for 10 min. Taking 1.6 mL from the beaker A added into the beaker B, quickly. After 5 sec, the beaker B was put into an ice bath to stop the reaction of the quantum dot solution. After 1 min, take it out to complete the preparation of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$:TOPO perovskite quantum dot precursor.

2.2 Procedure of LEDs

The LED structure in this work is deposited PEDOT:PSS hole injection layer, poly-TPD hole transporting layer, $\text{CsPbBr}_{1.2}\text{I}_{1.8}$:TOPO perovskite QD emitting layer, TPBi electron transport layer, and Ag anode on the ITO glass substrate with electrode pattern in this order. The PEDOT:PSS solution was deposited onto the ITO glass substrate with electrode pattern by spin coating at 5000 rpm for 60 s and baked at 120 °C for 20 min to form a hole injection layer. The ITO glass substrates with electrode pattern were washed in an ultrasonic cleaner with deionized water, acetone, and isopropyl alcohol for 30 min before to deposit the PEDOT:PSS films. The hole transporting layer was formed by spin-coating poly-TPD chlorobenzene solution (12 mg mL^{-1}) at 6000 rpm for 60 s and baking at 100 °C for 10 min. Sequentially, $\text{CsPbBr}_{1.2}\text{I}_{1.8}$:TOPO perovskite QD emitting layer was formed by spin coating at 2000 rpm for 60 s followed by annealing at 100 °C for 5 min. Finally, the 40-nm-thick TPBi electron transport layer and 100-nm-thick Ag anodes were deposited by thermal evaporation under a high vacuum of 5×10^{-6} Torr. The active area of the device was approximately 2 mm × 2 mm. After fabrication, the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ perovskite LEDs were encapsulated using UV glue and a glass cover in a glove box. The typical diagram of the perovskite quantum dot LED is shown in Figure 2.

2.3 Characterization

Transmission Electron Microscope (TEM) was employed to observed the particle size distribution of perovskite quantum dots (QDs). Morphologies of the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs were observed using a field-emission scanning electron microscope (FESEM, ZEISS Sigma, ZEISS, Munich, Germany). X-ray diffraction (XRD) patterns were characterize using X-ray diffractometer (X'Pert PRO MRD, PANalytical, Almelo, Netherlands). The photoluminescence (PL) spectra were recorded by fluorescence spectrophotometer (F-7000, Hitachi, Tokyo, Japan). The characteristic of LEDs, such as light output

power, electroluminescence, and current–voltage (I – V), were measured using PR-670 spectroradiometer and Keithley 2420 sourcemeter.

3. Results And Discussion

Figure 3 shows TEM images of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs mixed with various concentration of TOPO. Obviously, the size distribution of QD particles appeared barely change, but condensation and compact as the concentration of TOPO added into the solution of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs increases due to self-aggregation effect [22,23].

Figure 4(a) plots the photoluminescence (PL) spectra of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs mixed with various concentration of TOPO. The PL peak position of all samples is around 652 nm. With the increase of the concentration of TOPO, the intensity of PL spectrum increases. The spectrum of sample of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ mixed with 0.4 mmole TOPO exhibits the strongest intensity and narrowest FWHM (28 nm), respectively. It may be attributed to the more condense of QDs as concentration of TOPO increases, as shown in Fig. 3. Another factor is the reduction of surface defects of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs after passivation treatment with TOPO [24]. Figure 4(b) plots the absorbance of spectra of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs mixed with various concentration of TOPO. The absorbance spectra are cut-off at ~ 650 nm for all samples. It consists with the PL spectra.

Figure 5(a)-(d) display top-view FESEM images of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ films mixed with various concentration of TOPO. As shown in Fig. 5(a), the morphology of TOPO-free $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ film appears some holes and incomplete. However, a complete $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ film was formed when the 0.2 mmole TOPO was added into QDs solution. It may be contributed to the self-aggregation effect of TOPO, as shown in Fig. 5(c) and (d) [22,23]. Although the coverage and compact are improved by using TOPO, the height difference on the surface of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$:TOPO film is great owing to the reaction rate. In this work, $\text{Pb}(\text{NO}_3)_2$ was employed to improve the morphology and to smooth the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$:TOPO perovskite QDs film, as shown in Fig. 5(e), because $\text{Pb}(\text{NO}_3)_2$ can suppress and slow down the growth of perovskite QD film [25,26]. The surface passivation and growth rate reduction can promote the crystalline quality of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ perovskite QDs films. As shown in Fig. 5(f), the peak locations of XRD patterns mostly same. All samples exhibit a typical cubic phase, the high pure α -phase is obtained with two main peaks at 14.6° and 29.5° , corresponding to the (100) and (200) planes of cubic phase, respectively [27,28]. Obviously, the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$:TOPO (0.2 and 0.4 mmole) films show a decreased full width at half-maximum (FWHM) for the (200) peak in comparison with TOPO-free film. It suggests increased crystallite size and decreased surface defect [27,29], in well agreement with the PL spectra mentioned above. In addition, no peaks were newly formed, disappeared or shifted, implying that the perovskite crystal structure did not change during TOPO and $\text{Pb}(\text{NO}_3)_2$ passivation treatment.

Figure 6 shows the atomic force microscope (AFM) images of the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs film. R_a is the average roughness and R_z is the difference between the highest point and the lowest point on the surface

of film. For the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$:TOPO (0.2 mmole) film without surface passivation using $\text{Pb}(\text{NO}_3)_2$, Ra and Rz are 15.2 and 123.8 nm, respectively. However, for the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$:TOPO (0.2 mmole) film with surface passivation using $\text{Pb}(\text{NO}_3)_2$, Ra and Rz are 2.7 and 24.8 nm, respectively. The surface of the film with treatment of $\text{Pb}(\text{NO}_3)_2$ is relatively smooth.

Structure of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ perovskite QDs LED is shown in Fig 7. The function of ITO, PEDOT:PSS Poly-TPD, QDs, and TPBi is anode, hole transport layer, hole injection layer, active layer, and electron transport layer, respectively. Figure 8(a) sketches the band structure of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ perovskite QDs LED. Poly-TPD and TPBi layers can effectively confine carriers in the structure.

As shown in Fig. 8(b), the peak range of electroluminescence (EL) spectra of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ perovskite QDs LED is from 668 to 671 nm. The variation may be contributed to error of measurement or process. The EL spectrum has a red shift of 20 nm compared with the PL spectrum. It may be contributed to the heat caused by resistance of materials in the LED structures. As shown in Figure 8(c), the brightness of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs LED doped with TOPO increases with the concentration of TOPO doping due to surface passivation and reducing non-radiative recombination in the perovskite active layer [24]. However, excessive TOPO will result in a decrease in brightness. The $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs LED doped with TOPO: 0.2 mmole shows a better luminous brightness with a luminosity of 437.2 cd/m^2 (@9.5 V)

In the next phase, $\text{Pb}(\text{NO}_3)_2$ was employed to add into the solution of perovskite:TOPO to improve the morphology and uniformity of active layer according to the AFM images, as shown in Fig. 6. The current efficiency and luminosity is 0.175 cd/A and 502.7 cd/m^2 , respectively (@11 V). The improvement is caused by slow formation rate of perovskite active layer, such that reduction of leakage current [30-32]. As shown in Fig. 8(f), after 14-days burn-in test, the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ QDs LED with doped TOPO: 0.2 mmole and $\text{Pb}(\text{NO}_3)_2$ shows the best luminance owing to the surface passivation and smooth surface of the active layer.

Figure 9 shows CIE chromaticity diagram of $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ with (TOPO, $\text{Pb}(\text{NO}_3)_2$). The inset is the photo with 10 V bias in the dark. The coordinates of CIE chromaticity diagram are (0.738, 0.2905). It is very close to the boundary CIE chromaticity diagram. The advantage of this work is that red-light color is very pure due to good uniformity of the perovskite active layer. It is suitable for the application of display technology.

4. Conclusions

In this study, the $\text{CsPbBr}_{1.2}\text{I}_{1.8}$ perovskite QDs were synthesized by thermal injection method. TOPO was doped into the traditional combination of OA and OAm ligands with three different concentrations. According to the PL spectra, it can be observed that the wavelength intensity of QDs increases with increase of the addition amount of TOPO. The LED based on QD with TOPO (0.2 mmole) and $\text{Pb}(\text{NO}_3)_2$ showed much higher brightness than that of the control device. $\text{Pb}(\text{NO}_3)_2$ can improve the surface

passivation of the CsPbBr_{1.2}I_{1.8}:TOPO layers. The quality of the film is improved according to the results of TEM and AFM images. The surface of the film is flat and the transportation of the electron transport layer and the light-emitting layer is enhanced. The coordinate of CIE chromaticity diagram is very close to the boundary. The advantage of this work is that red-light color is very pure due to good uniformity of the perovskite active layer. In the future, it is suitable for the application of display technology.

Declarations

Declaration of interest

None declared.

Acknowledgments

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Figures

Figure 1

Process flowchart of CsPbBr_{1.2}I_{1.8}:TOPO perovskite quantum dots (QDs) precursor.

Figure 2

Diagram of CsPbBr_{1.2}I_{1.8} perovskite red LED structure.

Figure 3

TEM images of CsPbBr_{1.2}I_{1.8} QDs mixed with (a) 0, (b) 0.1 mmole, (c) 0.2 mmolw, and (d) 0.4 mmole TOPO.

Figure 4

Photoluminescence and absorbance spectra of CsPbBr_{1.2}I_{1.8} mixed with various concentration of TOPO.

Figure 5

Top-view SEM images and XRD pattern.

Figure 6

AFM images of CsPbBr_{1.2}I_{1.8} films with (a) TOPO film and (b) TOPO-Pb(NO₃)₂.

Figure 7

Cross-sectional SEM image of perovskite CsPbBr_{1.2}I_{1.8} QD LED.

Figure 8

Perovskite CsPbBr_{1.2}I_{1.8} QD LED device performance: (a) Band gap structure, (b) normalized EL spectra, (c) luminance characteristic, (d) current density characteristics, (e) external quantum efficiency (EQE), and (f) aging time results.

Figure 9

CIE chromaticity diagram of $\text{CsPbBr}_{1.2}\text{I}_{1.8}:(\text{TOPO}, \text{Pb}(\text{NO}_3)_2)$. The inset is the photo with 10 V bias in the dark.