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Fine-Tuned Multilayered Transparent Electrode for Highly Transparent Perovskite Light-Emitting Devices

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Abstract

The high photoluminescence quantum yield, wide color tunability and narrow bandwidth of perovskite nanocrystals make them favorable for light source and display applications. Here,

Conflict of Interest

The authors declare no conflict of interest.

highly transparent green-light-emitting devices (LEDs) using inorganic cesium lead halide perovskite nanocrystal films as the emissive layer are reported. The effect of multilayered nanostructured transparent electrode on optical properties and performance within the LEDs is investigated by fine tuning layer thickness. The results show that the light transmission in visible region can be enhanced with this nanostructured film. These LEDs exhibited a high transmittance (average 73% over 400–700 nm) and high brightness of 2640 and 1572 cd m⁻² for indium-doped tin oxide (ITO) cathode and MoO_x/Au/MoO_x anode sides, respectively.

Keywords

capping layer; charge injection; light-emitting device; perovskite nanocrystal; transparent

In recent years, perovskite materials have become a significant class of optoelectronic materials, widely used in photovoltaics,^[1] light-emitting devices (LEDs),^[2] and lasers.^[3] The excellent performances are mainly attributed to their unique optoelectronic properties, such as high optical absorption coefficient, direct band gap, long carrier-diffusion length, and high carrier mobility.^[4] Due to the enhanced stability and radiative recombination efficiency, the all-inorganic cesium lead halide (CsPbX₃, X = Cl, Br, and I) perovskite nanocrystals (NCs) have been developed as promising emitters.^[5] Notably, the photoluminescence (PL) quantum yields of these inorganic perovskite NCs are characterized up to 90%, also making them appealing for LED applications.^[6]

Transparent LEDs (TLEDs) are necessary in realizing new LED applications in many areas, such as integration into screens in electronic equipment or windows in automobiles, and may also be important for the fabrication of vertically stacked device geometry to obtain full color.^[7] Therefore, it is significant to investigate perovskite-based TLEDs for future applications. However, perovskite materials have high absorption coefficients, which will hinder the transmission of visible region photons, so the light-emitting layer should be as thin as possible to maximize the light pass through the whole device.

In order to fabricate TLEDs, transparent electrodes for both anode and cathode are required. Typically, indium-doped tin oxide (ITO)-coated glass has been used as transparent electrode for photovoltaic and LEDs because of the high transparency and conductivity.^[8] For the other side electrode, several kinds of materials have been developed, such as transparent conductive oxides,^[9] ultrathin metal films,^[10] metal nanowires,^[11] carbon materials,^[12] and conducting polymers.^[7b] In view of the high sensitivity of perovskite materials to temperature and solvent, ultrathin metal films deposited by thermal evaporation may be the most suitable alternative transparent electrode. However, there is significant light reflection at the metal–air interface because of the refractive index mismatch, leading to reduced transmission of the whole device.^[13] To overcome this disadvantage, a high refractive index material (such as MoO₃) can be deposited to reduce the light reflection.

Herein, we report transparent cesium lead tribromide (CsPbBr₃) NC-LEDs for the first time, with a multilayered MoO_x/Au/MoO_x (MAM) structure as a transparent electrode. Usually, MoO_x/Au film is used as anode in LEDs, where MoO_x aims to enhance hole injection. In this paper, an additional MoO_x layer was deposited, with the aim to enhance light

transmission. By fine-tuning the thickness of the MoO_x capping layer, the devices exhibited a high transparency, with an average transmittance of 73% over the visible range (400–700 nm). When applied a driving voltage, the device emitted bright green light, transmitting from both ITO cathode side and MAM anode side. The total peak brightness of the device was 4212 cd m^{-2} , while the maximum luminance and external quantum efficiency (EQE) for ITO and MAM electrode sides were 2640 cd m^{-2} and 0.35%, 1572 cd m^{-2} and 0.23%, respectively.

To investigate the optical properties, the absorption and PL spectra of the CsPbBr_3 NC solution were recorded at room temperature (Figure 1a, together with a photograph of the NC solution emitting bright green light). A sharp transition is observed at around 500 nm in the absorbance spectrum. The narrow PL spectrum of CsPbBr_3 NCs corresponds to excitonic emission is peaked at 517 nm, with a narrow full width at half maximum (FWHM) of 16 nm. The crystallinity of CsPbBr_3 NCs was characterized by X-ray diffraction (XRD). The XRD pattern shown in Figure 1b indicates the CsPbBr_3 NC powder belonging to a cubic phase. A representative transmission electron microscopy (TEM) image of CsPbBr_3 NCs (Figure 1c) shows the monodispersed cubic shape NCs with an average edge length of 10–11 nm.

Since CsPbBr_3 NCs intrinsically possess lower valence band maximum (-6.19 eV) energy level as compared with the highest occupied molecular orbital energy level of typical conjugated organic molecules or polymers ($\approx -5.5 \text{ eV}$), the hole injection was found to be difficult due to the large energy barrier.^[14] Herein, we adopted an inverted LED design, which enables the systematic engineering of the hole transport layer (HTL) with conventional organic materials. According to our previous work,^[15] the flatband energy level diagram of device can be mapped in Figure 2a. The emitting layer was sandwiched between the hybrid inorganic/organic charge-transport layers. The CsPbBr_3 NC-LEDs have a typical multilayer structure consisting of ITO (transparent cathode), ZnO/polyethylenimine (PEI) (electron transport layer), CsPbBr_3 NCs (emissive layer), 4,4'-bis(carbazole-9-yl)biphenyl (CBP), 4,4',4''-tris(carbazol-9-yl)triphenylamine (TCTA) (HTL), and MAM (transparent anode). ZnO NCs are widely employed in LEDs to transport electrons and block holes.^[16] Figure 2b shows the absorption spectrum of our ZnO NC solution, with a TEM image. The average size of our ZnO NCs is around 2 nm, which will lead to a higher lowest unoccupied molecular orbital (LUMO) energy, thus provide a more efficient electron injection within the device than larger sizes.

In the whole device, ZnO, CBP, and TCTA are all wide-band gap materials, with high transparency in visible region. Therefore, the perovskite NC layer and MAM electrode have important effect on the optical properties within the LEDs. Generally, the emissive layers in NC-LEDs are very thin, making them highly transparent. Therefore, the major factor affecting the light transmission of the device will be the top MAM electrode. One MoO_x layer can be used as a hole injection layer (HIL), which is common for inverted optoelectronic devices.^[16a] For the other MoO_x layer, it will enhance the light transmission and film conductivity as a capping layer.^[17]

As the device structure is electron abundant, hole injection will have large effect on device efficiency. According to the literature, MoO_x (5.44 eV work function) can shift the work function of Au electrode to higher values and as a result enhances the hole injection.^[17c,18] In order to design highly efficient TLEDs, the MoO_x layer thickness should be optimized. Figure 3 shows the performance of LEDs with an opaque electrode as a function of MoO_x layer thickness. It can be obviously seen that the device performances are strongly dependent on the thickness of MoO_x layer. First, the MoO_x layer will enhance hole injection, resulting in an improved device performance with 20 nm MoO_x . Second, when the thickness of MoO_x layer kept increasing, the device luminance, current efficiency, and device EQE all decreased. It may be caused by the increase of series resistance at the MoO_x -Au interface, as a result, hole-injection rate will slow down, leading to poorer device performances. By finely tuning the MoO_x layer, the best-performed LED with opaque electrode can be obtained, with a luminance of 4550 cd m^{-2} .

Electrodes with both high transparency and high conductivity are essential in order to realize effective transparent LEDs. As discussed above, we employed transparent ITO and an ultrathin Au film with 10 nm thickness as cathode and anode, respectively. To obtain details about the effect of MoO_x capping layer on the transparency of Au film, the resultant MoO_3/Au and MAM structures were prepared for optical transmission measurements, where the thicknesses of the first MoO_x layer and Au film were fixed at 20 and 10 nm, respectively. Figure 4a shows the optical transmission spectra of these films. It is obvious that the transmittance of Au film is significantly enhanced after capped with an additional MoO_x layer. Besides, the transmittance is strongly dependent on the thickness of the capping layer. The enhanced transmission can be attributed to decreased light reflection at the interface of Au/air. However, when the thickness of MoO_x capping layer kept increasing, the transparency of MAM films shifted to an opposite trend, which was resulted from stronger light absorption by the MoO_x capping layer. Therefore, it is significant to finely tune the thickness of MoO_x capping layer to maximize the transparency of MAM. After fine-tuning, the transparency of our electrode reached a highest value when the MoO_x layer thickness was about 25 nm. As shown in Figure 4b, the best MAM electrode exhibits a maximum transmittance of 77% (around 530 nm) and an average transmittance of 73% over the visible range (400–700 nm). We measured the resistance of the MAM film using the four-point probe method, and found the value was $13.3 \Omega \text{ sq}^{-1}$. As a result, the MAM films can be served as the device anodes due to their high transparency and conductivity. Also, the transmittance of an ITO film was measured over the visible range, and the average value was found to be around 93% (Figure 4b). For the whole device (including the glass substrate), a high transparency (58% around 520 nm) was obtained. A photograph of our transparent device is given in Figure 4b. The background image can be clearly seen through the transparent LED, demonstrating the device's high transparency.

In order to measure the emission performances from both bottom and top sides, we fabricated several devices simultaneously under control conditions. One was used for bottom emission measurement, while another one for top emission. Current density–voltage (J – V) and luminance–voltage (L – V) characteristics are shown in Figure 5a,b. Since the device's performances are not perfectly the same, there is a small difference between the two J – V curves in Figure 5a.

Noticeably, the maximum luminance of bottom (substrate) and top (MAM film) emissions was 2640 and 1572 cd m^{-2} , respectively. The total emission (the sum of bottom and top emission at the same voltage) reached a peak luminance of 4212 cd m^{-2} . To assess the reproducibility, 24 devices were fabricated, showing that the emitting luminance from bottom side was a little higher than that from the top side. The slightly lower luminance of top emission can be attributed to the relatively lower transmittance of MAM anode. Compared with the above device with opaque electrodes, the transparent devices worked at a little higher voltage. It is similar to some previous reports, which may be due to the higher resistance of the ultrathin Au film^[10a]. Also, the results are tabulated in Table 1 for easy comparison. As a consequence, the peak current efficiency and EQE for top side of the transparent LED was 0.82 cd A^{-1} and 0.23%, while the bottom side presented a slightly higher efficiency of 1.38 cd A^{-1} and 0.35%. Considering the practical application, the electroluminescence (EL) spectra of the transparent device were recorded when varying the emission angles, as shown in Figure 5e,f. Obviously, the EL spectra show no shift at peak position, which demonstrates the high transparency of the device. These results demonstrate that our transparent green LEDs can work efficiently and have a great potential for display.

Figure 6a shows the comparison of EL spectra from both MAM and ITO sides at an operating voltage of 7 V. The device's EL spectrum peaked at 518 nm with a FWHM of 18 nm, which is consistent with the NC solution PL spectrum with a 517 nm emission peak and a 16 nm FWHM as shown in Figure 1b. The EL emission through the top electrode is almost the same compared with the EL spectrum from the bottom. The symmetric emissions from both sides correspond with Commission Internationale de l'Eclairage (CIE) color coordinates of (0.12 and 0.73), as shown in Figure 6b. The inset of Figure 6a is a photograph of a transparent device emitting bright green light under a driving voltage of 7 V. In Figure 6c, the EL intensity of the ITO side and MAM side exhibited similar but a little faster decrease with the increasing emission angles compared with Lambertian patterns.

In conclusion, we successfully fabricated transparent green LEDs based on CsPbBr₃ NCs for the first time, with nearly Lambertian emission characteristics. These highly transparent LEDs employed inverted device structures, which helped to reduce the hole-injection barrier. The total emission of these devices achieved a peak luminance of 4212 cd m^{-2} . Full color transparent perovskite NC-LEDs could be fabricated simply by replacing CsPbBr₃ NC films with other perovskite NC emissive layers. Our study demonstrates the potential of perovskite nanomaterials for transparent display applications.

Experimental Section

Chemicals:

All chemicals were used directly. PbBr₂ (99.999%), Cs₂CO₃ (99.9%), and oleylamine (OLA, 80–90%) were purchased from Aladdin. Oleic acid (OA, 90%) was obtained from Alfa. 1-Octadecene (ODE, 90%), Zn(Ac)₂ (99.999%), and sodium hydroxide (99.99%) were got from Aldrich. Toluene and hexane were acquired from Aldrich.

Synthesis of CsPbBr₃ NCs:

The synthesis of CsPbBr₃ NCs was carried out as literature described.^[19] For the preparation of Cs-oleate precursor, a mixture of Cs₂CO₃ (0.8 g), oleic acid (OA, 2.5 mL), and 1-octadecene (ODE, 30.0 mL) was added into a three-neck flask. The flask was dried at 120 °C for 1 h and then heated to 150 °C until all Cs₂CO₃ was dissolved. For the synthesis of CsPbBr₃ NCs, ODE (10 mL) and PbBr₂ (0.138 g) were added into a 50 mL three-neck flask and dried at 120 °C under vacuum for 1 h. To dissolve PbBr₂, dried OA (1 mL) and oleylamine (OLA, 1 mL) were injected into the flask under N₂ to obtain a clear solution. The temperature was raised to 180 °C and 1 mL of Cs-oleate solution was injected quickly. After 5 s, the mixture was quickly cooled by an ice-water bath. The reaction mixture was directly centrifuged at 5000 rpm for 10 min; then the precipitate was collected and dispersed in 3 mL toluene. One more centrifugation for 10 min at 12 000 rpm was applied and the precipitate was dissolved in 2 mL of toluene for use.

Synthesis of ZnO NCs:

The synthesis of ZnO NCs followed a published method.^[20] A total of 0.4403 g Zn(Ac)₂ and 30 mL of ethyl alcohol were loaded in a 100 mL three-neck flask. After continuously flowing N₂ for 10 min, the mixture was then heated to boiling point temperature under a reflux condenser. When the mixture turned into clear, the heating mantle was then turned off and removed to cool the solution to room temperature naturally. When the solution temperature was stable, a solution consisting of 0.2 g sodium hydroxide and 10 mL ethanol was injected into the flask rapidly, and the solution was kept stirring for 4 h to grow NCs. To obtain pure products, excess hexane was used to precipitate NC products. Then the precipitate was dissolved in 3 mL ethanol. The purification process was repeated twice. Finally, the solution was filtered through a 0.45 µm filter for further use.

Device Fabrication:

For device fabrication, the ITO glass substrate was washed by deionized water, chloroform, acetone, and isopropanol in sequence and then treated by UV-ozone for 15 min. A ZnO film (≈40 nm) was deposited by spin-coating ZnO NC solution, and the prepared film was annealed at 100 °C for 10 min in air. The substrate was transferred into a glove box for the next layer. A solution of polyethylenimine (PEI) (dissolve in 2-methoxyethanol in 0.2% mass fraction) was spin coated onto the ZnO film at a speed of 3000 rpm and annealed at 110 °C for 10 min. CsPbBr₃ NC films (≈20 nm) were deposited by spin coating the prepared NC solution at 1000 rpm. The device was then transferred into a vacuum deposition chamber (1×10^{-7} torr) to deposit the CBP (≈10 nm), TCTA (≈35 nm), MoO₃, and Au (≈80 nm) layers by thermal evaporation.

Characterizations:

Optical absorption and transmission measurements were carried out in a PerkinElmer Lambda 950 spectrometer. Powder XRD patterns were collected on a Bruker SMART-CCD diffractometer. PL spectra were recorded by using a Cary Eclipse spectrofluorimeter. The current–voltage characteristics of the device were performed by Keithley 2612B sourcemeter. The luminance was recorded by using a calibrated Newport 1936-R power

meter with a 918D-SL-0D3R silicon photodetector. The EQE values were calculated using the modified coefficients since the emitting patterns were a little different to the Lambertian pattern. The EL spectra were measured via a Maya spectrometer (Ocean Optics) coupled to an optical fiber. TEM characterization was done on a FEI Tecnai F20 microscope.

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Figure 1.

a) The absorption and PL spectra of CsPbBr₃ NC solution, with an inset showing the photograph of the NC solutions under 365 nm irradiation; b) XRD pattern of the CsPbBr₃ NC powder; and c) TEM image of CsPbBr₃ NCs.

Figure 2. a) Device energy band diagram of the CsPbBr₃ NC-LED. b) The UV-vis absorption spectrum of ZnO NCs, with a TEM image.

Figure 3.

Comparison of a) current density–voltage ($J-V$) and b) luminance–voltage ($L-V$) characteristics of CsPbBr₃ NC-LED as a function of MoO_x layer thickness. c) External quantum efficiency and d) current efficiency versus current density characteristics of CsPbBr₃ NC-LED as a function of MoO_x layer thickness.

Figure 4.

a) Visible transmittance spectra of transparent electrode without any capping layers and with different thicknesses of the MoO_x capping layer; b) Transmittance spectra of the three samples with the structure of ITO, MAM, and the whole device, plus a photograph of the transparent LED.

Figure 5.

a) Current density–voltage (J – V) and b) luminance–voltage (L – V) characteristics of the transparent LED; c) current efficiency and d) external quantum efficiency versus current density characteristics of the LED; EL spectra of the LED for bottom side e) and top side f) with emission angles varying from 0° to 75° .

Figure 6.

a) EL spectra of the transparent device for both bottom and top sides and a photograph of a working LED at 7 V; b) CIE coordinates of the EL spectra for both bottom and top sides; and c) variations of EL intensity as a function of emission angles.

Table 1.

Device characteristics of the transparent NC-LEDs.

Side	V_{on} [V]	I_{max} [cd m ⁻²]	η_{EQE} [%] peak/at 100 mA cm ⁻²	η_p [lm W ⁻¹] peak/at 100 mA cm ⁻²	η_A [cd A ⁻¹] peak/at 100 mA cm ⁻²
Bottom	≈2.4	2640	0.35/0.33	0.91/0.47	1.38/0.28
Top	≈2.5	1572	0.23/0.21	0.60/0.31	0.82/0.78

^{a)} V_{on} represents the applied voltage when the device luminance reaches 1 cd m⁻².