Effect of Chloride Ion Concentrations on Luminescence Peak Blue Shift of Light-Emitting Diode Using Anti-Solvent Extraction of Quasi-Two-Dimensional Perovskite

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Abstract: In recent years, Perovskite Light-Emitting Diodes (PeLEDs) have received considerable attention in academia. However, with the development of PeLEDs, commercial applications of full-color PeLED technology are largely limited by the progress of blue-emitting devices, due to the uncontrollably accurate composition, unstable properties, and low luminance. In this article, we add Cesium chloride (CsCl) to the quasi-two-dimensional (quasi-2D) perovskite precursor solution and achieve the relatively blue shifts of PeLED emission peak by introducing chloride ions for photoluminescence (PL) and electroluminescence (EL). We also found that the introduction of chlorine ions can make quasi-2D perovskite films thinner with smoother surface of 0.408 nm. It is interesting that the EL peaks and intensities of PeLED are adjustable under different driving voltages in high concentration chlorine-added perovskite devices, and different processes of photo-excited, photo-quenched, and photo-excited occur sequentially with the increasing driving voltage. Our work provides a path for demonstrating full-color screens in the future.

Key words: Perovskite Light-Emitting Diodes (PeLEDs); quasi-two-dimensional (quasi-2D); chloride ions; blue shifts

1 Introduction

In recent years, as the development of light-emitting materials in Light Emitting Diodes (LEDs), metal halide perovskites have received considerable attention due to the high Photoluminescence Quantum Efficiency (PLQE), high mobility, adjustable colors, and easy process[1–5]. Both maximum External Quantum Efficiency (EQE) of green and red perovskite LEDs (PeLEDs) have exceeded 20% in just a few years[6–10]. Although great progress has been made in perovskite light-emitting devices, some problems still remain unsolved for PeLEDs in the blue light field, such as low efficiency, spectral fluctuations, and short life[11–13]. The process of preparing the PeLED for stable and high-efficiency blue light plays a key role in the application in the full-color display field.

Currently, bulky cations with long-chain ammonium groups are introduced based on three-dimensional perovskite to build a quasi-two-dimensional (quasi-2D)-layered perovskite with a structure of Multiple Quantum Wells (MQWs), which can emit blue light[14–20]. This quasi-2D perovskite is formed by a solution self-assembly method. It has a natural MQWs structure and good film forming properties. It is an effective strategy for making blue light PeLEDs.

In 2017, Cheng et al.[11] prepared a single halogen...
sky blue perovskite device with an MQWs structure by adding phenamine bromide (4-PBABr) to CsPbBr$_3$ at the first time, but the emission peaks were not pure. In 2019, Li et al.\cite{12} used three different proportions of chloride and bromide ions to prepare a three-dimensional perovskite structure and added phenethylammonium bromide (PEABr) to generate a quasi-2D structure, which emits blue light, but their experimental methods are relatively complicated and the device is unstable. In the same year, Wang et al.\cite{21} mixed phenylethylammonium chloride (PEACl) into cesium lead halide perovskites to produce a mixture of two-dimensional and three-dimensional perovskites and add yttrium (III) chloride to the mixture to further improve PLQE. However, it contains too many components and it is difficult to control the growth of the crystal structure.

It is reported in previous works that perovskite nanowires are synthesized through anion exchange reactions\cite{22}. In our article, we use quasi-2D perovskite film without CsCl to produce a bright green light. In addition, by mixing CsCl into the precursor solution, the quasi-2D material of perovskite is formed directly and the polycrystalline film successfully emits blue light. The electroluminescence (EL) of the classic LED of the “sandwich” structure based on the chlorine-added quasi-2D film is also significantly blue shift compared to the chlorine-free device. This phenomenon indicates that chloride ions can induce the blue shift of the emission peak of quasi-2D perovskite. The experimental method is flexible and convenient, which is the effective way of blue light generated by quasi-2D perovskite.

2 Experimental

2.1 Material
CsBr (>99.9%), PbBr$_2$ (>99.99%), PEABr (>99.5%), PEDOT:PSS 4083, LiF, CsCl (>99.9%), and TPBi were purchased from Xi’an Polymer Light Technology Corporation. Chlorobenzene (CB, 99.8%) were purchased from Shanghai Mater Win New Materials Co. Ltd., and dimethyl sulfoxide (DMSO, >99.0%) was purchased from Alfa Aesar. All substances were used as received.

2.2 Device manufacturing

2.2.1 Cleaning the indium tin oxide substrate
First, the patterned Indium Tin Oxide (ITO) conductive glass substrate was cleaned with acetone in ultrasonic vibration cleaners for 20 min. Then, the conductive glass substrate was cleaned with isopropyl alcohol for 20 min. After that, the conductive glass substrate was cleaned with ethanol for 20 min. In addition, the washed conductive glass substrate was purged with nitrogen. Finally, the cleaned conductive glass substrate was treated with a UV light cleaner for 20 min.

2.2.2 Hole injection layer fabrication

The hole injection layer was fabricated using aqueous PEDOT:PSS solution spin-coated onto the ITO substrate at 5000 rpm for 40 s, and then annealed at 150°C for 30 min on a hot plate to form a structurally stable film.

2.2.3 Manufacture of perovskite light-emitting film

The precursor solutions of perovskite prepared by properly dissolving stoichiometric PEABr, CsBr, CsCl, and PbBr$_2$ in DMSO under continuous agitation for 12 h at room temperature, and the required precursor solution was obtained by filtering.

Perovskite films were prepared by spin-coating the precursor solution at 3000 rpm for 120 s. The anti-solvent, chlorobenzene, was introduced at 40 s after the start of spin-coating to obtain a brighter quasi-2D perovskite film. The rotation coating process is shown in Fig. 1.

Fig. 1 Schematic procedure for manufacturing quasi-2D perovskite films by spin coating of precursor solutions and chlorobenzene.
2.2.4 Manufacture of electron injection layer and counter electrode

The electron injection layer is TPBi. TPBi (50 nm) and LiF/Al (3 nm/50 nm) electrodes were deposited using a thermal evaporation system under a high vacuum of $<4 \times 10^{-6}$ mbar (1 mbar = 100 Pa). The active area of the device was 0.105 cm$^2$, as defined by the overlapping area of the ITO and Al electrodes.

2.3 Characterization

The images of the Scanning Electron Microscopy (SEM) and the Atomic Force Microscope (AFM) were obtained with thicknesses and morphological characterization of the perovskite films. Photoluminescence (PL) spectrum of the perovskite films and the EL spectrum of the devices were recorded by a systematic measuring equipment from Ocean Insight.

3 Results and Discussion

In this article, we prepare precursor solutions for quasi-2D perovskites of different compositions by adding CsCl of different concentrations. The different compositions are shown in Table 1.

To investigate the effect of CsCl of different concentrations on the thickness and quality of quasi-2D perovskite films deposited on PEDOT:PSS films, measurements are made using the SEM and the AFM. As shown in Fig. 2a, without CsCl, the perovskite films are thicker. As CsCl concentrations increase, quasi-2D perovskite films become increasingly thinner (as shown in Figs. 2b and 2c) and can facilitate the formation of quasi-2D perovskites that emit blue light. When CsCl is introduced in quasi-2D perovskite, the most uniform perovskite films are prepared on the PEDOT:PSS film. This can be further confirmed by AFM measurements, in which the surface roughness $R_q$ decreases significantly from 2.12 nm to 0.408 nm, which may indicate that Cl$^-$ plays an important role in the preparation of high quality perovskite films with sufficiently smooth surfaces.

The results show that the introduction of Cl$^-$ can effectively decrease the roughness of the films and be conducive to the formation of more uniform and thinner

Table 1 Precursor solutions of perovskite for different compositions.

| Sample | PEABr (mmol) | CsBr (mmol) | PbBr$_2$ (mmol) | CsCl (mmol) | DMSO (mL) |
|--------|--------------|-------------|-----------------|--------------|-----------|
| 0 mmol CsCl | 0.33 | 0.33 | 0.5 | – | 1.0 |
| (66.70 mg) | (70.27 mg) | (183.5 mg) | – |
| 0.2 mmol CsCl | 0.33 | 0.13 | 0.5 | 0.2 | 1.0 |
| (66.70 mg) | (27.70 mg) | (183.5 mg) | (33.7 mg) |
| 0.4 mmol CsCl | 0.33 | 0.13 | 0.5 | 0.4 | 2.5 |
| (66.70 mg) | (27.70 mg) | (183.5 mg) | (67.4 mg) |

Fig. 2 Cross-sectional SEM and AFM images of the perovskite films. The roughness of the films is recorded on the left side of the images.
quasi-2D perovskite films.

The PL spectra of quasi-2D perovskite films are measured, as shown in Fig. 3a. The PL peaks of three different perovskite films correspond to 519 nm, 507 nm, and 494 nm. From Fig. 3a, we can conclude that quasi-2D perovskite films without CsCl show strong PL intensity with the highest emission peak at 519 nm. Although the PL intensity is reduced when the CsCl is added to perovskite precursor solutions spin-coated on PEDOT:PSS films, the position of the emission peak has a blue shift. A clear blue shift can be seen from the normalized PL spectra (Fig. 3b). With the increase in CsCl concentration, the position of the emission peak of the perovskite films moves from 519 nm to 507 nm. When CsCl is rich, it moves to 494 nm. It is concluded that some chloride ions introduced in the quasi-2D perovskite cause a blue shift in the position of the peak of emission. When calculating the Full Width at Half Maximum (FWHM), as shown in Table 2, it is seen that the introduction of 0.2 mmol of CsCl has the lowest FWHM and the highest luminous purity.

To discover the influence of CsCl of different concentrations on the EL characteristics of quasi-2D perovskite films, we manufacture PeLEDs consisting of glass/ITO/PEDOT:PSS/quasi-2D perovskite/TPBi/LiF/Al (Fig. 4a), where the quasi-2D perovskite is the light-emitting layer, apart from TPBi and LiF/Al that were deposited in a vacuum, the ITO was etched, other layers were solution processed via spin coating. The cross-sectional SEM image of the complete device is shown in Fig. 4b.

PeLED applications require different driving voltages. Therefore, there are still many challenges to achieve a highly stable blue PeLED. As shown in Fig. 5, it records that the stability of the quasi-2D perovskite LEDs with different concentrations of CsCl and the changes in the EL spectra with driving voltage. When the actuation voltage increases from 3 V to 8 V, the EL intensity of quasi-2D PeLED with 0 mmol CsCl increases correspondingly and reaches the highest

| Characterization | Sample      | FWHM (nm) |
|------------------|-------------|-----------|
| PL               | 0 mmol CsCl | 32.27     |
|                  | 0.2 mmol CsCl | 20.66   |
|                  | 0.4 mmol CsCl | 24.09   |
| EL               | 0 mmol CsCl | 24.21     |
|                  | 0.2 mmol CsCl | 24.68   |
|                  | 0.4 mmol CsCl | 43.85   |

Table 2 Precursor solution of perovskite for different compositions.
The EL intensity of the quasi-2D PeLED with 0.2 mmol CsCl increases first and then decreases, and reaches the highest intensity (Fig. 5b) at 7 V. The EL intensity of the quasi-2D PeLED with 0.4 mmol CsCl reaches its maximum value when the voltage is increased to 5.5 V and then decreases with additional voltage increase (Fig. 5c). As the voltage increases further to 8.5 V, the LED is excited again (Fig. 5d).

When the concentration of chloride ions in the precursor solution of perovskite is rich, the corresponding LED device sequentially displays the processes of photo-excited, photo-quenched, and photo-excited with the conduction voltage increasing from 0 V to 15 V (Fig. 6). As shown in Fig. 6, the black, blue, and red lines refer to the maximum intensity, luminance, and wavelength at the drive voltage, respectively.

The black and blue lines show the tendency to increase at first and then decrease at low voltage (4–6 V) and high voltage (8–15 V). This trend indicates that the LED has gone through two light emission stages between 0 V and 15 V. In the first light emission stage, both lines reach their maximum at 5.5 V, and the corresponding peak wavelengths are 504 nm on the red line. Obviously, the
maximum brightness appears at 11 V in the second stage of light emission (512 nm in the red line) and is different from the maximum intensity of the black line at 10.5 V. It is considered that the difference can be attributed to better capture the green light.

The red line (Fig. 6) shows that the peak wavelength appears at 4 V (500 nm). There is no light intensity with the voltage ranging from 6 V to 8 V, because the LED is in the extinguished state. When the driving voltage is greater than 12 V, the wavelength of the emission peak continues to increase and reaches to 518 nm at 15 V. It indicates the red shift of the luminescence peak with increasing voltage. We infer that there is a relatively blue peak due to the introduction of chloride ions. As the voltage increases, the phase separation is due to the instability of the crystal phases containing chloride ions, and the peak position of the green light is caused by the crystal phases that contain more bromide ions [12].

We placed the emission peaks of the three devices with maximum luminance at the coordinate of the Commission Internationale de l’Eclairage (CIE), as shown in Fig. 7. The 0 mmol CsCl device exhibits excellent color purity at green wavelengths, with CIE chromaticity coordinates at (0.13, 0.63). The 0.2 mmol CsCl device exhibits excellent color purity at light green wavelengths, with CIE chromaticity coordinates at (0.16, 0.47). The 0.4 mmol CsCl device exhibits excellent color purity at light blue wavelengths, with CIE chromaticity coordinates at (0.14, 0.42).

In Fig. 8a, the peaks around 515 nm, 508 nm, and 504 nm are assigned to the characteristic peaks of quasi-2D PeLEDs with 0 mmol, 0.2 mmol, and 0.4 mmol CsCl, respectively. In addition, the EL intensity decreases with the increasing of CsCl concentration. The FWHM extracted from the PL and EL spectra in Fig. 3a and 8a are calculated, respectively. As shown in Table 2, the FWHM for PL spectra reaches the minimum value of 20.66 nm, indicating that the highest luminous purity is obtained when 0.2 mmol CsCl is added to the quasi-2D PeLEDs. However, the FWHM for EL spectra increases as the CsCl concentration increases from 0 mmol to 0.4 mmol, showing deterioration in luminous purity.

It is clearly seen in Fig. 8b that the normalized EL spectra are similar to the normalized PL spectra in Fig. 3b, showing the slight blue shift.

The effect of voltage on luminance and the Luminance-Voltage (L-V) curves obtained (Fig. 8c) are investigated. It is clearly observed that the luminance of the quasi-2D PeLED without CsCl increases as the voltage increases from 3V to 8V, indicating that the device is stable. The intensity of 0.2 mmol CsCl added to quasi-2D PeLED reaches the maximum value when the voltage is increased to 7 V and then decreases with the increase in CsCl concentration, which indicates that a weaker non-radiative recombination has occurred. However, when the CsCl concentration is 0.4 mmol, the luminance of quasi-2D PeLED drops dramatically starting from a fairly low voltage of 5.5 V, indicating that a strong non-radiative recombination occurs, which leads to poor device stability.

The current density-voltage (I-V) curves of the device (Fig. 8d) are measured. From Fig. 8d, we can see that the device without CsCl shows a low injection current, indicating that the charge injection is balanced and the device is stable. However, the injection current of the device increases slightly when 0.2 mmol CsCl is added to quasi-2D PeLED, resulting in current leakage and imbalance in current injection, which indicates that a slight non-radiative recombination has occurred, being similar to the result in Fig. 8c. In addition, when the CsCl concentration is increased to 0.4 mmol, the injection current fluctuates to a very high value, indicating the strong non-radiative recombination in the quasi-2D PeLED, which is consistent with the L-V curves.

4 Conclusion

In this article, we can obtain thinner quasi-2D perovskite films with a smoother surface by controlling the concentration of CsCl in precursor solutions of quasi-2D perovskite. As the concentration of CsCl increases, the peak positions of the PL spectra show blue shifts. When
it increases to 0.2 mmol, the FWHM is the smallest (20.66 nm), indicating that it has the highest luminous purity. The EL spectra also show blue shifts, which is consistent with the result of the PL spectra. In contrast, the luminous purity becomes significantly attenuated with the increase in FWHM. When the voltage is applied to the device, PeLED without CsCl shows greater stability.

The stability of PeLED decreases with the increasing of chloride ions, which may be caused by the halide migration. However, PeLED with 0.4 mmol of CsCl still shows a higher emission peak at 5.5 V and provides a path for full-color display. An interesting process is found: The manufactured PeLED device sequentially exhibits the photo-excited, photo-quenched, and photo-excited process with an increase of the driving voltage when the chloride ion concentration of the perovskite precursor solution is high enough. It provides the basis for adjusting both the color and intensity by voltage in the future.

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