Core/Shell Perovskite Nanocrystals: Synthesis of Highly Efficient and Environmentally Stable FAPbBr₃/CsPbBr₃ for LED Applications

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Lead halide perovskite nanocrystals (PeNCs) are promising materials for applications in optoelectronics. However, their environmental instability remains to be addressed to enable their advancement into industry. Here the development of a novel synthesis method is reported for monodispersed PeNCs coated with all inorganic shell of cesium lead bromide (CsPbBr₃) grown epitaxially on the surface of formamidinium lead bromide (FAPbBr₃) NCs. The formed FAPbBr₃/CsPbBr₃ NCs have photoluminescence in the visible range 460–560 nm with narrow emission linewidth (20 nm) and high optical quantum yield, photoluminescence quantum yield (PLQY) up to 93%. The core/shell perovskites have enhanced optical stability under ambient conditions (70 d) and under ultraviolet radiation (50 h). The enhanced properties are attributed to overgrowth of FAPbBr₃ with all-inorganic CsPbBr₃ shell, which acts as a protective layer and enables effective passivation of the surface defects. The use of these green-emitting core/shell FAPbBr₃/CsPbBr₃ NCs is demonstrated in light-emitting diodes (LEDs) and significant enhancement of their performance is achieved compared to core only FAPbBr₃-LEDs. The maximum current efficiency observed in core/shell NC LED is 19.75 cd A⁻¹ and the external quantum efficiency of 8.1%, which are approximately four times and approximately eight times higher, respectively, compared to core-only devices.

1. Introduction

The newly emerged lead halide perovskite nanocrystals (PeNCs) have drawn extensive attention owing to their tunable optical properties, narrow emission bandwidth, and high photoluminescence quantum yield (PLQY) and were successfully used in a range of optoelectronic devices such as photodetectors and light-emitting diodes (LEDs). However, their inherent instability and degradation of the PLQY over time limit further advance of PeNCs in practical applications. Furthermore, ionic nature presents significant drawback as the PeNCs are extremely sensitive to environmental conditions, particularly to exposure to moisture, oxygen-rich atmosphere, and light. Currently intensive research efforts focused on development of synthesis methods and postsynthesis treatments aiming to enhance their stability by modification of crystal structure and chemical composition. Most promising approaches include embedding of PeNCs into a solid matrix and surface modification of PeNCs with more stable capping ligands, such as silica and alumina coating and polymer matrices. However, presence of these insulating matrices limits uses of the PeNCs in applications requiring charge transfer from the nanocrystals, such as LEDs and photovoltaic devices. Recently significant enhancement of the stability for all-inorganic cesium lead bromide perovskite (CsPbBr₃) was achieved by overgrowing the NCs with TiO₂ shell; however, the stability of organic–inorganic NCs remains to be addressed. Of particular interest is growth of optically active all-inorganic perovskite shell over organic-containing PeNCs, which has not yet been explored and could bring significant improvements to the optical stability of these NCs. The two materials, CsPbBr₃ and formamidinium lead bromide (FAPbBr₃), have different lattice structures with lattice mismatch of 3.4% presenting challenges for their use in core/shell structure.

Here we report on development of strategy for synthesis of monodispersed perovskite nanocrystals capped with all inorganic shell of CsPbBr₃ grown epitaxially on the surface of...
FAPbBr3 NCs. The growth of a layer of alloyed FA$_x$Cs$_{1-x}$PbBr$_3$ is used to address lattice mismatch between the core and the shell. After the formation of the core/shell structure, the surface of NC core is effectively passivated reducing the number of surface defects. By adjusting the synthesis conditions and composition of the nanocrystals we achieve stable PLQYs of up to 93% for FAPbBr$_3$/CsPbBr$_3$ core/shell NCs grown with molar ratio of Cs: FA = 1:1. The presence of CsPbBr$_3$ shell over FAPbBr$_3$ NCs leads to better environmental stability, with PLQY maintained at $\sim$ 80% of its initial value under long term (70 d) storage in ambient conditions. We also note that the core/shell NCs have remarkable stability under continuous irradiation with the ultraviolet (UV) light for at least 50 h, retaining QY of $\sim$75%. We demonstrate that these core/shell NCs can be used as emitters in LEDs with highly efficient electroluminescence performance. The achieved maximum current efficiency (CE) and external quantum efficiency (EQE) are 19.75 cd A$^{-1}$ and 8.1%, respectively. The results demonstrated here offer realistic prospects for utilization of core/shell perovskite NCs in optoelectronics.

2. Results and Discussion

Colloidal FAPbBr$_3$ NCs were synthesized by hot-injection method at $T = 130$ °C following a method adopted from ref. [23]. To overgrow the FAPbBr$_3$ NCs with shell of CsPbBr$_3$ we develop a facile wet-chemical approach, where different amounts of precursor materials (Cs$_2$CO$_3$, PbBr$_2$) and long-chain capping ligands (oleic acid (OA) and oleylamine (OLA)) and 1-octadecene (ODE) are injected into the solution of preformed FAPbBr$_3$ NCs. The resulting solution is heated to 80 °C to form NCs, retaining QY of $\sim$75%. We demonstrate that these core/shell NCs can be used as emitters in LEDs with highly efficient electroluminescence performance. The achieved maximum current efficiency (CE) and external quantum efficiency (EQE) are 19.75 cd A$^{-1}$ and 8.1%, respectively. The results demonstrated here offer realistic prospects for utilization of core/shell perovskite NCs in optoelectronics.

The blueshift of the PL emission peaks in the core–shell NCs is distinctly different from that of CsPbBr$_3$ NCs (the two samples are prepared under the same reaction condition and with the same raw stoichiometric analysis).[29] We also note that the PL emission of FAPbBr$_3$/CsPbBr$_3$ core–shell NCs is distinctly different from that of CsPbBr$_3$ NCs (the two samples are prepared under the same reaction condition and with the same raw stoichiometric ratio, see Section SI1 and Figure S4, Supporting Information). For alloyed FAPbBr$_3$/CsPbBr$_3$ NCs we observe significant blueshift and broadening of the PL spectra compared to core–shell NCs. The blueshift of the PL emission peaks in the core–shell NCs is attributed to the formation of alloyed layer of FA$_x$Cs$_{1-x}$PbBr$_3$ at the core–shell interface of NCs, resulting in reduced effective...
Meanwhile, the long-range, infinite lattice periodicity of a core NC is disturbed during the shell coating, and the differences of dielectric constant and absorption coefficient between the core and shell also affect the physical properties of the PeNCs. Hence, we conclude that with the increasing Cs content, the all-inorganic shell of CsPbBr$_3$ is formed over the transitional alloyed layer. The observed blueshift is consistent with increasing strength of exciton confinement in FAPbBr$_3$ for NCs capped with a thicker shell of CsPbBr$_3$. Importantly, by overgrowing the NCs with inorganic shell we increase the PLQY reaching a maximum value of 93% for $X = 1.0$ (inset in Figure 3a). In order to understand the dynamics of carrier recombination, we study the time-resolved PL (Figure 3b). The decay times can be fitted well-with a biexponential function (Equation (1))

\[
A(t) = A_1 \exp\left(-\frac{t}{\tau_1}\right) + A_2 \exp\left(-\frac{t}{\tau_2}\right)
\]

(Figure 2. Morphological and structural properties of core FAPbBr$_3$ NCs and FAPbBr$_3$/CsPbBr$_3$ core/shell NCs ($X = 1.0$). a) High-angle annular dark field-scanning transmission electron microscopy (HAADF-STEM) image and b) corresponding elemental mapping image showing the elemental distribution of Pb and Br for FAPbBr$_3$ NCs. c) HRTEM image of a typical FAPbBr$_3$ NC and corresponding selected area electron diffraction (SAED) pattern (inset). d) HAADF-STEM image and e) corresponding elemental mapping image showing the elemental distribution of Pb, Br, and Cs for FAPbBr$_3$/CsPbBr$_3$ NCs. f) HRTEM image of a typical FAPbBr$_3$/CsPbBr$_3$ NC and corresponding SAED pattern (inset). g,h) Size distribution analysis for the samples in (a) and (d), respectively. i) XRD patterns of FAPbBr$_3$ NCs, CsPbBr$_3$ NCs, and a series of thickness-controlled FAPbBr$_3$/CsPbBr$_3$ core/shell NCs (The values of $X$ are the different molar ratio of Cs/FA).
where $A_1$ and $A_2$ are constants, $t$ is time, and $\tau_1$ represents the trap-assisted recombination at grain boundaries and $\tau_2$ is ascribed to radiative recombination (see Table S1, Supporting Information). We find that with increasing $X$, corresponding to increasing shell thickness, the PL lifetime of FAPbBr$_3$/CsPbBr$_3$ NCs exhibits the same trend as that of PLQYs, with $\tau$ value increased from 30.2 ns of FAPbBr$_3$ NCs to 40.1 ns of FAPbBr$_3$/CsPbBr$_3$ NCs (at $X = 1.0$). These observations confirm that the shell overgrowth provides more efficient passivation of surface defects on FAPbBr$_3$ NCs hence reducing the number of nonradiative recombination centers.

The long-term stability of the NCs is of paramount importance for their application in optoelectronic devices. Hence, we examine the shelf life of the core/shell NCs. We find that storage of the NCs in ambient conditions (air and room temperature) (Figure 3c) leads to significant decrease of the relative PLQY of FAPbBr$_3$ NCs to $<10\%$ over 20 weeks, while FAPbBr$_3$/CsPbBr$_3$ NCs retain PLQY $>60\%$. The core/shell NCs also demonstrate enhanced stability under UV illumination ($\lambda = 365$ nm), retaining over 80$\%$ of original PLQY, compared to $<20\%$ for core only NCs, following 50 h exposure (Figure 3d). Overall, we found that the stability of PeNCs with core/shell structure is significantly improved compared to bare NCs.

Formation of core/shell perovskite NCs was further confirmed by our temperature dependent PL studies (80–300 K) (Figure 4a,b). With increasing temperature, we observe a blueshift of PL peak position by 74 meV for FAPbBr$_3$ NCs with gradients of 0.3 meV K$^{-1}$, characteristic for this type of materials.$^{[33,34]}$ In contrast, the PL peak of FAPbBr$_3$/CsPbBr$_3$ core/shell NCs blueshifts by $\approx30.6$ meV, with gradient of 0.3 meV K at $T < 150$ K and 0.6 meV K$^{-1}$ at higher temperature. The observed difference of temperature dependence of the optical energy gap confirms presence of a shell providing stronger exciton confinement. The $T$-dependent blueshift of optical bandgap is common for perovskite NCs and was observed for CH$_3$NH$_3$PbX$_3$ and Cs$_3$Sb$_2$Br$_9$ PeNCs.$^{[35,36]}$ With increasing $T$, the integrated PL intensity decreases due to the thermally activated nonradiative recombination processes (Figure 4c). We estimated the exciton binding energy ($E_b$) from a temperature-dependent PL spectra by following equation$^{[37]}$

$$I(T) = \frac{I_0}{1 + A \exp\left(\frac{-E_b}{K_B T}\right)}$$

(2)

where $I_0$ is the emission intensity at low temperature, $A$ is a constant, and $K_B$ is the Boltzmann constant. From our curve fitting we find $E_b = 51$ meV for FAPbBr$_3$ NCs and $E_b = 64$ meV for FAPbBr$_3$/CsPbBr$_3$ core/shell NCs. The observed increase of $\approx130\%$ confirms stronger binding of the exciton in core/shell NCs. The optical linewidth (Figure 4c) of the FAPbBr$_3$/CsPbBr$_3$ NCs is FWHM $= 115$ meV at RT, which is significantly broader compared to core-only FAPbBr$_3$ NCs (90 meV), which we attribute to a decrease of the strength of exciton-phonon coupling for core/shell NCs.$^{[36]}$

Figure 3. a) UV–vis absorbance and PL spectra and b) time-resolved PL decay curves of FAPbBr$_3$ NCs and FAPbBr$_3$/CsPbBr$_3$ NCs with different thickness-controlled (The values of $X$ are the different molar ratio of Cs/FA and other values are the corresponding quantum yields). Time dependence of relative PLQY of core only and core/shell NCs c) under storage in ambient conditions and d) under exposure to 365 nm UV light.
Enhanced optical properties and environmental stability of the core–shell NCs could offer exciting prospects for their exploitation in optoelectronic devices. Hence, we produce LED structures with a layer of FAPbBr₃ NCs or FAPbBr₃/CsPbBr₃ core/shell NCs. (Figure 5a,b, see also Section SI1 and Table S2, Supporting Information). The perovskite LED (PeLED) consists of multilayers of an indium tin oxide (ITO) anode, poly(3,4-ethylenedioxythiophene)-polystyrene sulfonate (PEDOT:PSS) hole injection layer (HIL), poly(N,N'-bis(4-butylphenyl)-N,N'-bis(phenyl)-benzidine) (poly-TPD) hole transport layer (HTL), a PeNCs emission layer, 1,3,5-tris(1-phenyl-1H-benzimidazol-2-yl) benzene (TPBI) electron transport layer, LiF electron injection layer, and Al cathode. Poly-TPD is used as an HTL and electron-blocking interlayer simultaneously due to its low electron affinity (≈2.2 eV) and high hole mobility, which is matched to that of TPBI (both around 1 × 10⁻⁴ cm² V⁻¹ s⁻¹).[38,39] Figure 5c shows normalized electroluminescence (EL) spectra of PeLEDs based on FAPbBr₃/CsPbBr₃ core/shell NCs with different values of X. We observe a blueshift of EL peak position from 536 to 508 nm with X increasing from 0 to 1.5, similar to that observed for PL peak (see Section SI2 and Figure S4, Supporting Information). We note that the separation between EL and PL peak positions decreases with increasing X, likely due to lower defect density, which is consistent with observed increase of PLQY.

We also examine the effect of washing on device performance (see Table S3, Supporting Information). We find the highest value of EQE with two repeats of washing for device fabricated with both core and core–shell NCs. Notably, up to ten-time increase in the EQE is observed for devices based on core–shell NCs. Hence we select the NCs with best performance for further studies. The current density–voltage (J–V), luminance–voltage (L–V), and current efficiency–current density (CE–J) curves of these devices are shown in Figure 5d–f. The device based on core-only FAPbBr₃ NCs has a luminance of 1593 cd m⁻² and CE of 4.80 cd A⁻¹. With increasing shell thickness (X = 0.25, 0.5, and 1.0) of core/shell NCs, the CE and EQE gradually increase to 19.75 cd A⁻¹ and 8.1% (X = 1.0), respectively. However, further increase of X to 1.5 leads to a decrease of the luminance of PeLEDs from 1758 to 957 cd m⁻² and the CE has reduced to 7.76 cd A⁻¹ (see also Table 1). We observe the maximum CE and EQE for the NCs with X = 1.0 and these values are fourfold and eightfold higher compared to those of the bare FAPbBr₃-based PeLEDs. We also noted that the PeLEDs based on core/shell NCs show a greatly improved operational lifetime compared with the FAPbBr₃-based PeLEDs due to the better optical stability of the NCs (see Figure S5, Supporting Information). The device parameters achieved in our work with FAPbBr₃ NCs overgrown with inorganic shell are better or comparable to those recently reported for hybrid perovskite LEDs.[40,41] These results confirm that the core/shell NCs can be used as an effective emitting layer in high performance PeLEDs with additional benefit of long shelf life.

3. Conclusion
In conclusion, we have developed an epitaxial growth method for the overgrowth of CsPbBr₃ shell on the surface of FAPbBr₃ NCs. The formed core/shell FAPbBr₃/CsPbBr₃ NCs have improved PLQY (93%) and long-term shelf life and optical
stability under UV illumination. We attribute the enhanced optical and environmental stability to effective passivation of the surface defects and hence increase radiative recombination. We demonstrated that the PeLEDs based on the FAPbBr3/CsPbBr3 core/shell NCs (Cs/FA = 1.0) have improved performance compared to those fabricated with core-only NCs, with maximum CE of 19.75 cd A⁻¹, EQE of 8.1%, which is almost 4.1-fold, 7.9-fold higher than that of the bare FAPbBr3-based PeLEDs. Notably, this work can open new avenues for the NC design and is relevant for their utilization in high-efficiency luminescent flat panel display applications.

4. Experimental Section

Synthesis of Perovskite Nanocrystals: All reagents were purchased and used without further purification: FA-acetate (Sigma-Aldrich), Pb(CH3COO)2 × 3H2O (Sinopharm Chemical Reagent Co., Ltd., ≥99.5%), octadecene (ODE, Sigma-Aldrich, 90%), OA (Sigma-Aldrich, 90%), OAmBr (Xi’an Polymer Light Technology Crop), toluene (Sinopharm Chemical Reagent Co., Ltd., ≥99.5%), PbBr2 (Sigma-Aldrich, 90%), Cs2CO3 (Sigma-Aldrich), OLA (Sigma-Aldrich, 90%). FAPbBr3 NCs were synthesized as following method reported in ref. [23]. Briefly, FA-acetate (0.078 g, 0.75 mmol), Pb(CH3COO)2 · 3H2O (0.076 g, 0.2 mmol), OA (2 mL, vacuum dried at 120 °C), and ODE (8 mL, vacuum-dried at 120 °C) were combined in a 100 mL and dried for 30 min under vacuum at 50 °C. The mixture was heated to 130 °C

Table 1. Performance parameters of FAPbBr3/CsPbBr3 core/shell based LEDs prepared with NCs with different molar ratio, X, of Cs:FA.

| Molar ratio FA: Cs, X | Tpeak [cd m⁻²] | CEpeak [cd A⁻¹] | EQEpeak [%] | Von [V] |
|----------------------|----------------|-----------------|-------------|--------|
| 0.25                 | 1281           | 2.89            | 0.59        | 2.9    |
| 0.5                  | 1528           | 5.22            | 1.29        | 2.8    |
| 1.0                  | 1758           | 19.75           | 8.10        | 2.6    |
| 1.5                  | 957            | 7.76            | 3.18        | 3.0    |
| 0                    | 1593           | 4.80            | 1.03        | 2.5    |
under N₂ atmosphere and OAmBr (0.21 g, 0.6 mmol) in toluene (2 mL) was injected. After 10 s, the reaction mixture was cooled on ice-water bath. The green solution of FAPbBr₃ NCs was collected and stored at T = 4 °C.

For synthesis of FAPbBr₃/CsPbBr₃ nanocrystals, PbBr₂ (0.1101 g, 0.3 mmol) and Cs₂CO₃ (0.0326 g, 0.1 mmol) were loaded into a 100 mL flask. Then, dried OA (1 mL), dried ODE (5 mL), dried OLA (0.5 mL), and as synthesized FAPbBr₃ nanocrystals (12 mL) were added. The mixture was heated to 80 °C for 20 min under N₂ atmosphere, followed by cooling on ice bath. The color of the reaction solution has changed significantly compared to the original solution of FAPbBr₃ NCs. Finally, the resulting solution was centrifuged to remove undispersed residue and aggregated nanocrystals. The FAPbBr₃/CsPbBr₃ NCs dispersed in hexane were stored in the dark at T = 4 °C.

Purification: As-prepared crude solution of FAPbBr₃ and FAPbBr₃/CsPbBr₃ NCs was put into centrifuge tubes and a certain volume of ethyl acetate was added (keeping the volume ratio of crude solution to ethyl acetate 1:3), then centrifuged at 7000 rpm for 5 min. After that, the precipitate was redissolved in n-hexane and centrifuged for 1 min at 5000 rpm. Finally, the supernatant was collected and stored at 4 °C. This is the PeNC solution after a purification, according to the actual test requirements or application needs, which can be purified twice or three times (see Table S3, Supporting Information).

Characterization Methods: Transmission electron microscope Talos F200X was used to assess the morphology, size distribution, and elements composition of the NCs. XRD patterns were recorded on a Bruker D8 Advance diffractometer with Cu Kα radiation range from 10° to 60° at a scanning rate of 4°·min⁻¹. UV-vis absorption spectra were measured on Hitachi J-3900H spectrometer. The photoluminescence (PL) spectra and time-resolved fluorescence were measured on Edinburgh FLs920 spectrometer. Temperature dependence of PL was measured on a Princeton Instruments Acton SP2750.

Device Preparation: PEDOT: PSS (Clevios PVP Al 4083) was purchased from Heraeus. Poly-TPD, TPBI, and LiF were purchased from Luminescence Technology and used without further purification. For device fabrication, the ITO glass was cleaned in ultrasound bath with detergent followed by deionized water, acetone, and isopropyl alcohol (30 min in each solvent). After drying, the substrates were treated in oxygen plasma for 15 min. PEDOT: PSS solutions (filtered through a 0.22 µm filter) were spin coated onto ITO glass at 4000 rpm for 40 s. Then perovskite NCs were spin coated at 4000 rpm for 60 s. After that, the substrates were transferred into a nitrogen glove box. The poly-TPD solution in chlorobenzene (8 mg mL⁻¹) was sequentially deposited by thermal evaporation at a base pressure of 4 × 10⁻⁶ Pa. The current density–voltage–luminance (J–V–L) characteristics were measured using Keithley 2400 sourcemeter.

Conflict of Interest
The authors declare no conflict of interest.

Keywords
core/shell nanocrystals, light-emitting diodes, optical stability, perovskite nanocrystals, quantum efficiency

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Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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