



Article Photodetector Based on CsPbBr₃/Cs₄PbBr₆ Composite Nanocrystals with High Detectivity

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Abstract: High-quality, all-inorganic CsPbBr₃/Cs₄PbBr₆ composite perovskite nanocrystals (NCs) were obtained with all-solution-processing at room temperature, and a photodetector (PD) with high detectivity was realized based on CsPbBr₃/Cs₄PbBr₆ NCs. The detectivity (*D**) of the proposed PD is 4.24×10^{12} Jones under 532 nm illumination, which is among the highest levels for PDs based on all-inorganic perovskite NCs. In addition, a high linear dynamic range (LDR) of 115 dB under 1 V bias was also realized. Furthermore, the underlying mechanism for the enhanced performance of the proposed PD was discussed. Our work might promote the preparation of high-performance PDs based on dual-phase all-inorganic perovskite nanocrystals.

Keywords: all-inorganic lead halide perovskite; CsPbBr₃; Cs₄PbBr₆; perovskite nanocrystals; photodetector

1. Introduction

All-inorganic lead halide perovskites have shown excellent photoelectrical properties with high absorption coefficients, wide spectra, high carrier mobility values, and long charge diffusion lengths. Because of these advantages, they are widely used in PDs [1–4], solar cells [5–8], lasers [9–11], light-emitting diodes [12–14], and so on. In addition, compared with the organic-inorganic hybrid perovskite, all-inorganic lead halide perovskites have higher chemical stability to the environment because there are no organic ions in them. Among many kinds of all-inorganic lead halide perovskites, CsPbBr₃ NCs demonstrate strong competitiveness for their excellent photoelectric characteristics, such as adjustable photoluminescence (PL) with narrow full-width at half maximum (FWHM) in visible light, an easy preparation process for their devices, and small size which causes a quantum size effect. Moreover, the CsPbBr₃ NCs can be stored in air for 30 days with no obvious decrease in their photoelectric performance [15]. Some research on optoelectronic devices based on CsPbBr₃ NCs has been conducted. For example, Li et al. reported an interesting recyclable dissolution-recrystallization phenomenon of CsPbBr₃ NCs and its application on the room temperature self-healing of compact and smooth carrier channels for PDs with D^* of 6.1 \times 10¹⁰ Jones [16]. Zeng et al. demonstrated a high-performance self-actuation PD, for which CsPbBr₃ NCs were synthesized using room temperature saturation recrystallization, and the NC films were prepared by a centrifugal casting method. Compared with the dripcoating method, the photocurrent of the device increased three-fold, and the optimized device had a high on/off ratio (>10⁵) [15]. However, the D^* of these all-inorganic perovskite NC PDs is not high enough. Generally, the detectivity can be improved by increasing the bright current and suppressing the dark current. Additionally, improving the quality of nanocrystalline films to reduce defect states is vital for the suppression of the dark current.



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Quan et al. have found that embedding CsPbBr₃ NCs in the Cs₄PbBr₆ matrix can prevent the agglomeration of CsPbBr₃ NCs [17]. This means that the presence of Cs₄PbBr₆ NCs could improve the quality of NC films. Thus, we prepared CsPbBr₃/Cs₄PbBr₆ composite NCs by controlling the proportion of PbBr₂ in the synthesis process. Cs₄PbBr₆ is an indirect band gap semiconductor with a wide band gap (3.8 eV) [18], and Cs₄PbBr₆ NC is a zerodimensional material, which absorbs light yet does not emit it [19]. This non-luminous property can effectively reduce radiation recombination. In addition, it has been proved that the presence of Cs₄PbBr₆ will enhance the absorption of the dual-phase perovskite NCs in the UV spectrum [17,20,21]. This is helpful for improving the detection performance of the PD in the UV spectrum.

Taking the above-mentioned issues into account, the saturation recrystallization method was utilized to synthesize the dual-phase NCs at room temperature. The dual-phase NC film was spin-coated on the customized substrate and Au electrode. Moreover, the planar metal-semiconductor-metal (MSM) PDs based on CsPbBr₃/Cs₄PbBr₆ composite NCs were realized. The proposed PD exhibited a high D^* of 4.24×10^{12} Jones, with an LDR of 115 dB under 1 V bias. To explain the improvement of D^* , the role of Cs₄PbBr₆ NCs was investigated. Our work might provide a guidance for developing a high-performance PD based on CsPbBr₃/Cs₄PbBr₆ perovskite NCs.

2. Materials and Methods

Materials: Cesium bromide (CsBr, 99.5%), lead bromide (PbBr₂, 99.0%), Oleic acid (OA, 85%) and oleylamine (OAm, 80–90%) were purchased from Aladdin. Toluene (AR) was purchased from Beijing Chemical Works. All these chemicals were used as received.

Synthesis of CsPbBr₃/Cs₄PbBr₆ NCs: The saturation recrystallization method was utilized to synthesize the NCs at room temperature without any noble gases, and the specific synthesis steps are shown in Figure 1. Firstly, the beaker and the small glass bottle were each cleaned with deionized water, isopropanol, and absolute ethanol for 15 min, and then dried with high-purity nitrogen. After that, they were placed on a hot table at 80 °C to accelerate solvent evaporation and ensure no residual water. Then, 0.4 mmol of CsBr and 0.4 mmol of PbBr₂ were dissolved in 10 mL of DMSO. A clear and transparent solution was obtained by ultrasonic cleaning, and no water entered the solution in this process. Then, 0.5 mL of OAm and 1 mL of OA were added as ligands of NCs to prepare a precursor solution. After that, 1 mL of the above precursor solution was dribbled into the 10 mL of toluene solution with a fast speed of 800 r/min. In this way, various inorganic ions were transferred from the benign solvent to the undesirable solvent, and the NCs were deposited due to the much higher solubility of NCs in DMSO than in DMSO mixed with toluene.



Figure 1. Schematic diagram of the fabrication process of the PD based on CsPbBr₃/Cs₄PbBr₆ NCs.

Fabrication of PD: The fabrication of the device was mainly based on the gold interdigital electrode and the inorganic perovskite NCs. The schematic diagram of the fabrication process is illustrated in Figure 1. The interdigital space of the gold interdigital electrode is 3 μ m and the thickness of the Au electrode is about 100 ± 10 nm. The substrate is made up of silicon (Si) wafer, SiO₂, and chromium (Cr) plating. A 300 nm-thick SiO₂ layer covers the surface of the silicon wafer. Additionally, there is a Cr plating layer with a thickness of 10 nm between the SiO₂ layer and the Au electrode. The planar PD was fabricated in a glove box by spin-coating. The NC solution was spin-coated on the substrate with the rotational speed of 2000 r/min. The device was then placed on a hot platform at 90 °C to vaporize the methylbenzene and DMSO.

Characteristics of nanocrystals: The photoelectric property of NCs was tested by a light-emitting diode (LED) light source (375 nm, Thorlabs, Shanghai, China). The optical image of NCs was characterized using an optical microscope (Nikon, LV150, Shanghai, China). The high-resolution transmission electron microscopy (HRTEM) images of NCs were collected using a JEOL JEM-2100 microscope (Nippon Electronics Corporation, Shanghai, China) at an accelerating voltage of 100 kV. The X-ray diffraction (XRD) spectrum of the NCs was measured by a diffractometer (Haoyuan Instrument Co., Ltd., DX-2700, Dandong, China). The absorption spectrum of NCs was determined by an ultraviolet-visible absorption spectrometer (Shimadzu, UV-2600, Shimadzu, Japan). The photoluminescence spectrum at room temperature was carried out by a home-built fluorescence spectrophotometer system using a 343 nm femtosecond laser (Light Conversion, Carbide 5W, Vilnius, Lithuania) as the excitation light source.

Photoelectric characteristics: The PD was placed on the probe table (Prcbe, Mini), and the external shielding box was used to isolate the external optical signals throughout the test. The current–voltage (I–V) curves and the transient photo responses of the PD based on inorganic perovskite NCs were tested using a semiconductor analyzer (Agilent, B1500, Shanghai, China). For completing the I–V measurements, the light intensity was tuned by adjusting the LED light source (375 nm, Thorlabs, Shanghai, China) at a fixed driving voltage. The transient photo responses were acquired with an LED lamp, which emitted light faster than the response of the measured PDs.

3. Results and Discussion

3.1. Composition of NCs

To determine the composition of NCs, XRD measurements were carried out, as shown in Figure 2a. The peaks at 15.185°, 21.551°, 30.644°, 34.371° and 37.767° can be indexed to the (100), (110), (200), (210) and (211) lattice planes, respectively, which are the characteristic peaks of the CsPbBr₃ phase. On the other hand, the peaks at 12.885°, 20.079°, 22.413°, 25.427°, 27.510°, 28.603° and 30.268° can be identified as the (110), (113), (300), (024), (131), (214) and (223) reflections, respectively, which are the characteristic peaks of Cs₄PbBr₆ [18,21]. In addition, the intensity of the diffraction peaks of Cs₄PbBr₆ is much higher than those of CsPbBr₃, which means that the main component of the NCs is Cs₄PbBr₆. To further investigate the composition of the NCs, the HRTEM images were obtained and shown in Figure 2b, and the selected area corresponding to Figure 2b is illustrated in Figure 2c. It can be found that the lattice fringes of 0.68 nm can be indexed to the (110) lattice planes of orthorhombic Cs₄PbBr₆, while the lattice fringes of 0.29 nm can be indexed to the (200) lattice planes of cubic CsPbBr₃. Thus, the dual-phase was also evidenced by the selected area TEM of dual-phase NCs.





Figure 2. (a) XRD patterns of the dual-phase CsPbBr₃/Cs₄PbBr₆ NCs; (b) HRTEM image of the dual-phase inorganic perovskite NC film, inset: Fast Fourier transform (FFT) image of the dual-phase inorganic perovskite NC film; (c) HRTEM corresponding to Figure 2b.

3.2. Optical Property of NCs

In order to investigate the optical property of NCs, its absorption and PL spectrum were tested, as shown in Figure 3a,b. We found that there were two absorption peaks in the absorption spectrum. One peak was located at 505 nm, and the other one was located at 316 nm. According to the existing research on CsPbBr₃, we know that the absorption peak at 505 nm is attributed to CsPbBr₃. Thus, it can be inferred that the absorption peak at 316 nm was caused by Cs₄PbBr₆. In addition, the absorption band edges of CsPbBr₃ and Cs₄PbBr₆ NCs are 360 nm and 547 nm, respectively. Based on this, the band gaps of CsPbBr₃ and Cs₄PbBr₆ NCs were calculated, which were 2.3 eV and 3.8 eV, respectively. This is in agreement with the literature [18]. For the PL spectrum (shown in Figure 3b), a narrow emission peak was observed at 520 nm with a FWHM of 26 nm, which is 3.1 nm lower that of pure CsPbBr₃ NCs (29.1 nm) [22]. This suggests that the quality of NC films was improved by the introduction of Cs₄PbBr₆ NCs. It is worth noting that there was only one PL peak that originated from CsPbBr₃ NCs, which is consistent with the previous studies' finding that Cs_4PbBr_6 does not emit light [23]. Moreover, the PL peak was in the green light emission band, which echoes the luminescence of the sample shown in the inset of Figure 3b. $CsPbBr_3/Cs_4PbBr_6$ NCs in toluene (illustration) were yellow under sunlight. However, when exposed to ultraviolet light, the CsPbBr₃/Cs₄PbBr₆ NCs in toluene emitted bright green light. This is also consistent with the reported emission band of CsPbBr₃/Cs₄PbBr₆ NCs [21].



Figure 3. (a) Absorption spectrum of CsPbBr₃/Cs₄PbBr₆ NCs; (b) PL spectrum of CsPbBr₃/Cs₄PbBr₆ NCs, insets show NC solution illuminated by a fluorescent lamp and a 375 nm LED lamp, respectively.

3.3. Performance of the Composite NC PD

The dual-phase inorganic perovskite NC film was spin-coated on the gold interdigital electrodes, and the planar MSM PDs were prepared. The I-V curves of the proposed PD under 375 nm LED illumination with different light intensities are shown in Figure 4a. It can be found that the photocurrent increased with the rise in the optical power density. Moreover, the dark current at 1 V bias was 6.67 pA, which was significantly suppressed compared with that of the PD based on pure CsPbBr₃ NCs (1.5 nA at 2 V bias) [15]. It suggests that the quality of NC film was improved by the introduction of Cs₄PbBr₆ NCs, which could prevent the agglomeration of CsPbBr₃ NCs. The light current with a light intensity of 10.2 mW/cm² at 1V bias was 19.32 nA and the ratio of light current to dark current was 2894 at 1 V bias. This value was much lower than that of the PD based on $CsPbBr_3 NCs (10^5 under 4.65 mW/cm^2 at 2 V bias)$, which meant that the bright current was weakened by the introduction of Cs_4PbBr_6 NCs. The external quantum efficiency (η_{EOE}) , responsivity (*R*), and *D*^{*} of the proposed PD were calculated and the results are shown in Figure 4c–e. As the power density of the incident light increased from $13 \,\mu W/cm^2$ to 2689 mW/cm², the $\eta_{\rm EOE}$ decreased continuously. At the lowest detectable illumination of 13 μ W/cm² at a bias of 1V, the η_{EOE} was only 22.1%. This value is also lower than the PDs based on $CsPbBr_3 NCs$ (41%) [16]. These phenomena may be due to the fact that the main component of dual-phase NC film is Cs₄PbBr₆. Moreover, the wide band gap of Cs₄PbBr₆ determines that the absorption wavelength of Cs₄PbBr₆ must be lower than 360 nm. However, due to the limitation of the experimental conditions, our test wavelength could only reach 375 nm. Thus, the light absorption intensity and the amount of photogenerated carriers of the PD based on CsPbBr₃/Cs₄PbBr₆ composite NCs were correspondingly reduced under the illumination of 375 nm. This also led to low R, which was only 0.094 A/W under the optical power of 13 μ W/cm². The expression of R was $\frac{l_L - l_d}{P_L - S}$ (I_L is photocurrent, I_d is dark current, P_{in} is incident optical power density, S is effective area). Although the PD had an ultra-low dark current, the light current was not satisfied, which led to a small R. However, compared with the previous reported MSM PDs based on CsPbBr₃ NCs (summarized in Table 1), the D^* of the proposed PD was among the highest level, which reached 4.24×10^{12} Jones under the optical power of 13 μ W/cm². Due to the great improvement of D*, it could be expected that the LDR would also be improved. Here, the LDR of the proposed PD under 1 V bias with 532 nm continuous laser illumination were shown in Figure 4b. It can be found that the proposed PD was capable of detecting incident light as low as 13 μ W/cm² under 1 V bias, and its LDR was 115 dB.



Figure 4. (a) *I*–*V* curves of CsPbBr₃/Cs₄PbBr₆ NC PD with different optical power densities under the irradiation of a 375 nm LED lamp; (**b–e**) LDR, η_{EQE} , *R*, and *D** measured using a 532 nm laser as a light source; (**f**) Change trend of the dark current after taking the logarithm of the x-axis and y-axis, the red line is the fitting curve.

Table 1. Summary of the performance of inorganic perovskite NC PDs.

Nanocrystal	R (mA/W)	η _{EQE} (%)	D* (Jones)	T _{rise} /T _{fall} (ms)	Ref.
CsPbI ₃	-	-	-	24 / 29	[23]
CsPbBr ₃	0.18	41	$6.1 imes10^{10}$	1.8 / 1.0	[16]
CsPbCl ₃	1890	-	-	41 / 43	[24]
CsPbBr ₃	4.71	16.69	4.56×10^{8}	0.2 / 1.3	[5]
$CsPbBr_3/Cs_4PbBr_6$	0.094	22.1	4.24×10^{12}	10.85 / 2.85	Our work

In order to explain the reason for the improvement of D^* , the type of contact between the semiconductor and the metal electrode was analyzed. Due to the high work function of Au, it can be expected that the interface between the inorganic perovskite NCs and the Au electrode will form a good ohmic contact [15]. This prediction was proved by the experiment. The *I*–*V* curve at different biases were measured, and Figure 4f shows the change trend of the dark current after taking the logarithm of the x-axis and y-axis. It is clear that the current increased significantly with the increase in bias, and the slope (n_1) of the curve is 1.07, which is almost linear. This means that a good ohmic contact was formed between the dual-phase NCs and Au electrode, providing a guarantee for the rapid transfer of the carrier. On the other hand, the band gaps of CsPbBr₃ and Cs₄PbBr₆ were 2.3 eV and 3.8 eV, respectively, and both the VBM and CBM of Cs₄PbBr₆ were higher than those of CsPbBr₃. Thus, by combining CsPbBr₃ NCs with Cs₄PbBr₆ NCs, the energy band of the composite NCs would be moved up as a whole relative to the Au electrode. Moreover, the height difference between the VBM of the composite NCs and the Fermi energy of the Au electrode would be reduced, which facilitates the transmission of electrons. This is also helpful for the improvement of D*. In addition, both the strongly suppressed dark current and the narrowed FWHM of the PL spectrum proved that the quality of the NC film was indeed greatly improved with the introduction of Cs₄PbBr₆ NCs. Furthermore, the radiation recombination could also be suppressed due to the non-luminous property of Cs₄PbBr₆ NCs. All these behaviors led to the improvement of D*.

To study the photoresponse of the composite NC PD, the transient response performance under 375 nm with a light intensity of 10.2 mW/cm² at 1 V bias were measured (shown in Figure 5a), which indicated that the proposed PD could respond stably when the illumination was turned on and off. Moreover, response speed was another important index of the photodetector, and the transient photocurrent response relationship (*I*-*T*) curve of the proposed PD under 1 V bias is shown in Figure 5b. According to the definitions of the rise time (T_{rise}) and fall time (T_{fall}) for the time when the photocurrent increased from 10% to 90% (declined from 90% to 10%) in the on and off cycles under light, T_{rise} and T_{fall} were 10.85 ms and 2.25 ms, respectively. The sum of the rise time and the fall time was considered the response time of the PD, which was 13.1 ms.



Figure 5. (a) Photocurrent–time response measured under 375 nm with a light intensity of 10.2 mW/cm^2 ; (b) Rise and fall time of the proposed PD.

4. Conclusions

In summary, we have successfully demonstrated all-solution-processed PDs based on CsPbBr₃/Cs₄PbBr₆ composite NCs. A good ohmic contact was formed between the gold electrode and CsPbBr₃/Cs₄PbBr₆ composite NCs, which provided a guarantee for the rapid transfer of the carrier. In addition, both the strongly suppressed dark current and the narrowed FWHM of the PL spectrum proved that the quality of the NC film was greatly improved with the introduction of Cs₄PbBr₆ NCs. Moreover, the nonluminous property of Cs₄PbBr₆ NCs could inhibit the radiation recombination. Thus, the *D** of the PD based on CsPbBr₃/Cs₄PbBr₆ composite NCs was improved, reaching 4.24×10^{12} Jones. Our work might provide guidance for developing a high-performance PD based on CsPbBr₃/Cs₄PbBr₆ perovskite NCs. **Author Contributions:** Conceptualization, R.W. and L.F.; formal analysis, G.L. and Y.H. (Yuying Hao); funding acquisition, W.W., T.J., G.L., Y.H. (Yuying Hao) and Y.C.; investigation, Y.H. (Yue Han) and Y.C.; methodology, F.Z. and T.J.; project administration, R.W. and L.F.; resources, Y.C.; supervision, Y.C.; validation, Y.H. (Yue Han) and L.S.; writing–original draft, Y.H. (Yue Han); writing–review and editing, R.W. and L.F. All authors have read and agreed to the published version of the manuscript.

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