



Sequential deposition method fabricating carbon-based fully-inorganic perovskite solar cells

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ABSTRACT Hybrid organic-inorganic halide perovskite material has been considered as a potential candidate for various optoelectronic applications. However, their high sensitivity to the environment hampers the actual application. Hence the technology replacing the organic part of the hybrid solar cells needs to be developed. Herein, we fabricated fully-inorganic carbon-based perovskite CsPbBr₃ solar cells *via* a sequential deposition method with a power conversion efficiency of 2.53% and long-time stability over 20 d under ambient air conditions without any encapsulation. An evolution process from tetragonal CsPb₂Br₅ to CsPb₂Br₅-CsPbBr₃ composites to quasi-cubic CsPbBr₃ was found, which was investigated by scanning electron microscopy, X-ray diffraction spectra, UV-vis absorption spectra and Fourier transform infrared spectroscopy. Detailed evolution process was studied to learn more information about the formation process before 10 min. Our results are helpful to the development of inorganic perovskite solar cells and the CsPb₂Br₅ based optoelectronic devices.

Keywords: perovskite solar cell, phase transition, carbon cathode

INTRODUCTION

In the past several years, hybrid organic-inorganic halide perovskites with the common formulation ABX₃ (where A is an organic cation, B is commonly Pb²⁺ and X is a halide) have undergone breathtaking progress in power conversion efficiency (PCE) from nascent 3.8% [1] to a striking record of 22.1% [2]. Hybrid organic-inorganic halide perovskites have been verified to be in possession

of many excellent properties, such as long electron/hole diffusion length [3], remarkable sunlight absorption [4], easy and low-cost fabrication process [5], etc., which make them an ideal candidate for lots of applications in photodetectors [6], light-emitting devices [7], semiconductor lasers [8], and solar cells [9–11].

However, the actual application of hybrid perovskites is hindered because of their strong sensitivity to ultraviolet (UV) light [12], heat [13], and moisture [14,15]. Fortunately, inorganic cesium lead halides, CsPbX₃ (X=Cl, Br, I) show prominent compositional stability under thermal stress and remarkable photoelectric features with high electron mobility up to ~1000 cm² V⁻¹ s⁻¹ [16]. In 2015, the first inorganic CsPbBr₃ perovskite solar cells (PSCs) were reported by Kulbak *et al.* [17], acquiring a PCE of about 5%. More recently, Liao *et al.* [18] reported CsPbX₃ nanowires stable solar cells with PCE of 1.21% and stability around 5500 h. Liang *et al.* [19] reported carbon-based CsPbBr₃ solar cells with a PCE of 6.7% and stability over three months in humid air (90%–95% relative humidity, 25°C). As for CsPbI₃, undesired orthorhombic phase was formed with a low PCE of 0.09% due to its poor phase stability to moisture [20]. CsPb₂Br₅ is another important material for optoelectronic applications. The advantages of CsPb₂Br₅ used in optoelectronic devices lie in the intrinsic sandwiched structure and physical properties from the structure. In the tetragonal CsPb₂Br₅ structure, one layer of Cs ions is sandwiched between two layers of Pb-Br coordination polyhedrons [21]. Now the applications of CsPb₂Br₅ include laser and

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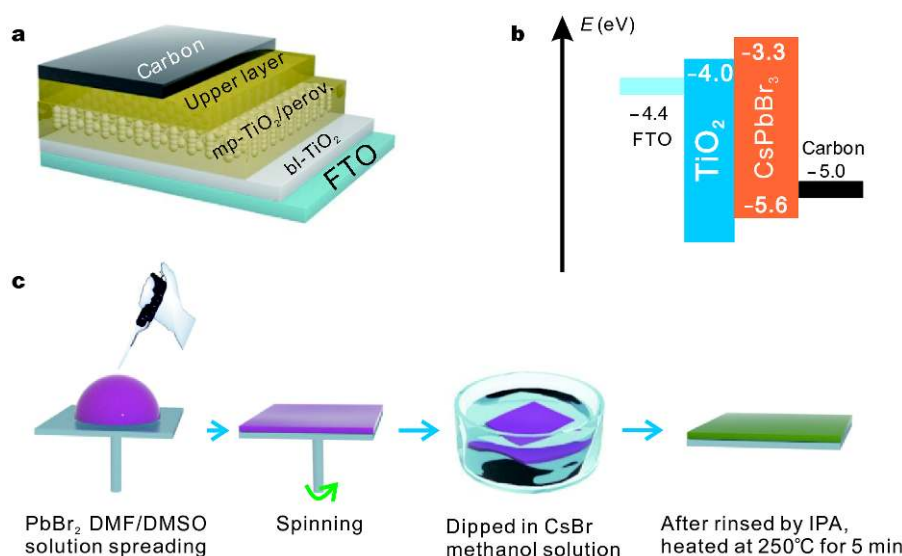


Figure 1 (a) Device architecture of CsPbBr₃/carbon based all-inorganic PSCs (glass/FTO/bl-TiO₂/ms-TiO₂-perovskite nanocomposite layer/perovskite upper layer/carbon); (b) energy level diagram of CsPbBr₃/carbon based all-inorganic PSCs; (c) scheme view of fabrication process of the perovskite film using sequential deposition method.

light-emitting devices [22–24]. Compared with the perovskite CsPbBr₃, the available research on the structure and physical properties of the tetragonal CsPb₂Br₅ is not very sufficient and many great blanks need to be filled [21]. Our results offer a facile way to synthesize CsPb₂Br₅, which could enlighten other researchers to develop and design more applications of CsPb₂Br₅. At the same time, we use sequential deposition method obtaining CsPbBr₃ films using carbon as hole-transporting material and photocathode, achieving a PCE of 2.53% with long-term stability. Scanning electron microscopy (SEM), X-ray diffraction spectra (XRD), Fourier transform infrared spectroscopy (FT-IR) and UV-vis absorption spectra were used to investigate CsPb₂Br₅, CsPbBr₃ and the transition from CsPb₂Br₅ to CsPbBr₃. Our results are helpful to the development of fully-inorganic CsPbBr₃ solar cells and CsPb₂Br₅ based optoelectronic device.

EXPERIMENTAL SECTION

Materials

F-doped SnO₂ glass (FTO, 15 Ω/square) was purchased from Pilkington TEC. Lead bromide (PbBr₂, ≥98%), *N,N*-dimethylformamide (DMF) and dimethyl sulphoxide (DMSO) were obtained from Aldrich. Cesium bromide (CsBr, >99%) was purchased from TCI. TiO₂ (particle size: about 30 nm, crystalline phase: anatase) was purchased from Dyesol. Methanol (AR) and isopropanol (IPA, 99.7%) were obtained from Sinopharm Chemical

Reagent Co., Ltd. All chemicals were straightly used without further purification.

Device fabrication

Etched FTO-coated glass substrates were cleaned by ultrasonication in deionized water followed by washed with acetone and ethanol. These substrates were exposed to UV-ozone for 15 min. A ~60 nm-thick TiO₂ blocking layer was deposited on the cleaned FTO by spray pyrolysis, using N₂ as carrier gas, at 430°C from a precursor solution of 0.6 mL of titanium diisopropoxide and 0.4 mL of bis(acetylacetonate) in 7 mL of anhydrous isopropanol. A mesoporous TiO₂ layer (particle size: about 30 nm, crystalline phase: anatase, diluted to *w/w*=1/5.5 in ethanol) about 100–150 nm thickness film was deposited by spin coating at 5000 rpm for 30 s onto the blocking layer (bl)-TiO₂/FTO substrates. After spin coating, the substrate was immediately dried on a hotplate at 105°C, and the substrates were then heated at 500°C to remove organic components followed by baking for 2 h. Then, as illustrated in Fig. 1c, firstly, 1.4 mol L⁻¹ PbBr₂ DMF/DMSO solution (DMF/DMSO *v/v*=9/1) was spread over the entire surface of the substrate. Secondly, the spin-coater was accelerated to the desired rotational speed at 2000 rpm for 30 s. Thirdly, the as-prepared PbBr₂ films were dipped into 15 mg mL⁻¹ CsBr methanol solution for various time. Finally, the perovskite films were rinsed by IPA and heated at 250°C for 5 min. Then, the carbon paste was deposited on the perovskite films by screen

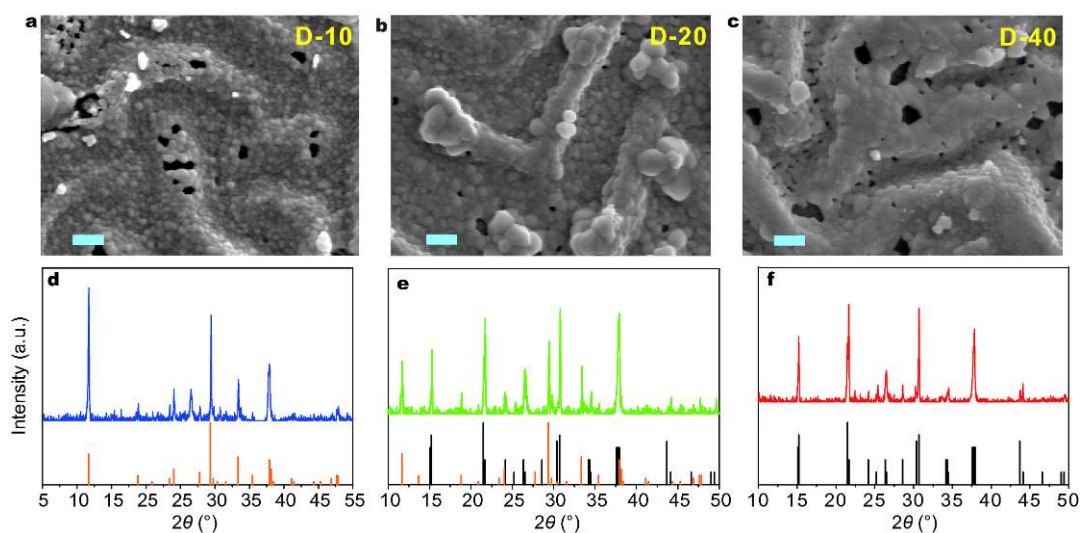


Figure 2 SEM Images of inorganic active layer: (a) D-10; (b) D-20; (c) D-40; the scale bar is 1 μm and XRD patterns of D-10 (d), D-20 (e), D-40 (f) with standard XRD patterns, orange line: tetragonal CsPb_2Br_3 , JCPDS No. 00-025-0211; black line: monoclinic CsPbBr_3 , JCPDS No. 00-018-0364.

printing followed by heating at 70°C for 1 h.

Characterization

Film morphology was investigated by using a high-resolution scanning electron microscope (S4800, Hitachi) equipped with a Schottky Field Emission gun. The X-ray diffraction spectra were recorded on an X'pert PRO X-ray diffractometer. The data were gathered at room temperature in the 2θ range 5°–50°. The J - V curves were measured using a solar simulator (Newport, Oriol Class A, 91195A) with a source meter (Keithley 2420) at 100 mA cm^{-2} illumination AM 1.5 G and a calibrated Si-reference cell. The J - V curves for all devices were measured by masking the active area with a black mask 0.09 cm^2 . The applied bias voltage was from 1.2 to -0.1 V and the scan rate was 50 mV s^{-1} . UV-vis absorption spectra were recorded on a UV-vis spectrometer (Hitachi U-3300). Fourier transform infrared spectroscopy (FT-IR, ThermoFisher IS50R, USA) was used to collect the spectral data for various solution in the range of 4000–500 cm^{-1} .

RESULTS AND DISCUSSION

Structure and morphology characterization

Fig. 1 presents the device architecture, energy level diagram as well as scheme of films fabrication process. The cell structure (Fig. 1a) exhibits a bilayer structure making up of mesoporous and planar structures, which is effective for adequately absorbing light and gathering charges.

Atop the mesoporous (mp)- TiO_2 layer lies a uniform perovskite active layer. Upward the perovskite active layer lies a layer of carbon working as bi-functional films for both electron blocking and hole collecting for the reason of its fit work function (-5.0 eV) close to that of gold (-5.1 eV). Fig. 1b exhibits the energy band diagram of FTO, TiO_2 , CsPbBr_3 and carbon layers, uncovering the smooth electron injection from CsPbBr_3 conduction band to TiO_2 conduction band and hole extraction from CsPbBr_3 valence band to carbon photocathode. Fig. 1c shows the perovskite films fabrication process, in which the as-prepared PbBr_2 films was dipped in 15 mg mL^{-1} CsBr methanol solution for 10 (D-10), 20 (D-20) and 40 min (D-40).

The surface morphology evolution of the inorganic active layer based on different dipping time was investigated by SEM (Fig. 2a–c). The films dipped in CsBr methanol solution for 10 min (D-10) exhibit relatively low surface coverage and small grain size (the average grain size is 216 nm calculated by software Nanomeasurer with 30 sample points.) The D-20 film is relatively more uniform with average grain size of 275 nm. For the D-40 film, the average grain size becomes larger up to 391 nm. Our results show that longer dipping time is beneficial for better performance of the CsPbBr_3 PSCs. It is worthy to note that all the three kinds of films have lots of fluctuation, which is detrimental to electron and hole transfer. The cause of the fluctuation is unclear and need to be further studied. Fig. S1 (Supplementary information) illustrates the carbon photocathode, which shows it com-

prised carbon nanoparticles with average diameter of ~100 nm.

The XRD measurement was conducted to investigate the crystal phase evolution of these samples. As depicted in Fig. 2d, the XRD pattern of D-10 films could match well with standard patterns of tetragonal phase CsPb_2Br_5 (JCPDS No. 00-025-0211), whose lattice parameters are $a=b=8.483 \text{ \AA}$, $c=15.250 \text{ \AA}$, $\alpha=\beta=\gamma=90.000^\circ$. Along with the prolonging of dipping time, as shown in Fig. 2e, a different material and phase emerged in the D-20 films, which is ascribed to monoclinic phase CsPbBr_3 (JCPDS No. 00-018-0364), with lattice parameters $a=b=5.827 \text{ \AA}$, $c=5.891 \text{ \AA}$, $\alpha=\beta=90.000^\circ$, $\gamma=89.650^\circ$. It is quite similar as cubic phase with slight difference of c and γ , so we call this structure as quasi-cubic phase CsPbBr_3 . In the D-20 films, the XRD peaks belong to two kinds of materials (tetragonal CsPb_2Br_5 and quasi-cubic CsPbBr_3), indicating the material and phase evolution is in progress and the CsPb_2Br_5 has not completely evolved to CsPbBr_3 . However, when we turn to the D-40 films, as illustrated in Fig. 2f, all the CsPb_2Br_5 converts to CsPbBr_3 . It is worthy to note that no peak of PbBr_2 could be found, indicating the sufficient reaction of PbBr_2 and CsBr with no PbBr_2 residual remained in all the three kinds of films. Fig. S2 shows absorbance of the D-10, D-20 and D-40 films. The light absorption peaks of D-20 and D-40 films are shorter than ~540 nm and the absorption peak of D-10 films is shorter than ~375 nm, which suggests tetragonal CsPb_2Br_5 was not suitable for photovoltaic applications.

To learn more information about the formation process at the interface of PbBr_2 films and the CsBr methanol solution before 10 min, we investigated the as-prepared PbBr_2 films dipped in the 15 mg mL^{-1} CsBr methanol solution for 1 (D-1), 2 (D-2) and 5 min (D-5). XRD measurement and UV-vis spectra were used to further investigate CsPb_2Br_5 . As depicted in Fig. 3a, the XRD patterns of D-1, D-2 and D-5 have slight difference in the intensity, while all matching well with the standard XRD pattern (CsPb_2Br_5 JCPDS No. 00-025-0211), indicating that even early at 1 min, the tetragonal CsPb_2Br_5 was formed at the interface. It is worthy to note that the full width at half maxima (FWHM) of XRD patterns of D-1, D-2 and D-5 films are wider than that of D-10, showing smaller grain size compared with the D-10 films according to Debye-Scherrer formula. As presented in Fig. S3, the light absorption peaks of these films are shorter than ~375 nm.

Combining the previous reports [21,22,25] and the fact that no PbBr_2 residual could be found in D-10 films, we hypothesize the reaction route according to the equation:

$\text{CsBr}+2\text{PbBr}_2\rightarrow\text{CsPb}_2\text{Br}_5$. Then, CsPb_2Br_5 continues to react with CsBr in methanol according to the equation: $\text{CsPb}_2\text{Br}_5+\text{CsBr}\rightarrow2\text{CsPbBr}_3$. In the first step, the effect of the $\text{PbBr}_2\cdot\text{DMF}$ adduct is considered to be important for the formation of CsPb_2Br_5 . Without the existence of the adduct, the reaction process should be different [19,26]. FT-IR was conducted to confirm the existence of the adduct. As depicted in Fig. 3b, the stretching vibrations of C=O in DMF and DMSO ($v/v=9:1$) mixed solution (T-1 solution) were located at 1670 cm^{-1} . When 513.8 mg PbBr_2 was added to 1 mL T-1 solution (T-2 solution), some of the vibrations of C=O shifted to 1638 from 1670 cm^{-1} , which could prove the existence of $\text{PbBr}_2\cdot\text{DMF}$ adduct.

Photovoltaic performance

The photovoltaic application of perovskite has recently become a hotspot. In consideration of this, we fabricated the PSCs based on D-10, D-20, D-40 films. Table 1 summarizes the typical photovoltaic properties of the three kinds of solar cells. Unfortunately, the device based on D-10 films did not perform photovoltaic properties because of its broad band gap. However, we propose a new strategy to synthesis CsPb_2Br_5 here, which will help other researchers to expand its application in other fields. The devices based on D-20 films show a relatively low open circuit voltage (V_{oc}), short current density (J_{sc}), PCE, and fill factor (FF) comparing with the devices based on D-40 films because of the existence of CsPb_2Br_5 . The best cell based on D-40 films, as depicted in Fig. 3c, yielded a J_{sc} of 3.52 mA cm^{-2} , a V_{oc} of 1.15 V, an FF of 0.624 and a PCE of 2.53%. Fig. 3d presents the cross-sectional SEM image of the device based on D-40 films, signifying a structure making up of FTO, 60 nm-thick bl-TiO_2 , 200 nm-thick meso- TiO_2 /perov., 650 nm-thick upper layer and carbon photocathode.

Stability of devices

The stability of solar cells is of fundamental importance to the commercialization process. Stability is still a challenge for the hybrid PSCs [27]. Herein the stability test of the devices based on D-40 films were conducted under ambient air without any encapsulation. Fig. 3e illustrates normalized stability curves of the device based on D-40 films, which shows that the PSCs were stable after 20 d. Prominent stability will be beneficial to the commercial application process.

CONCLUSION

In summary, we proposed a simple sequential deposition

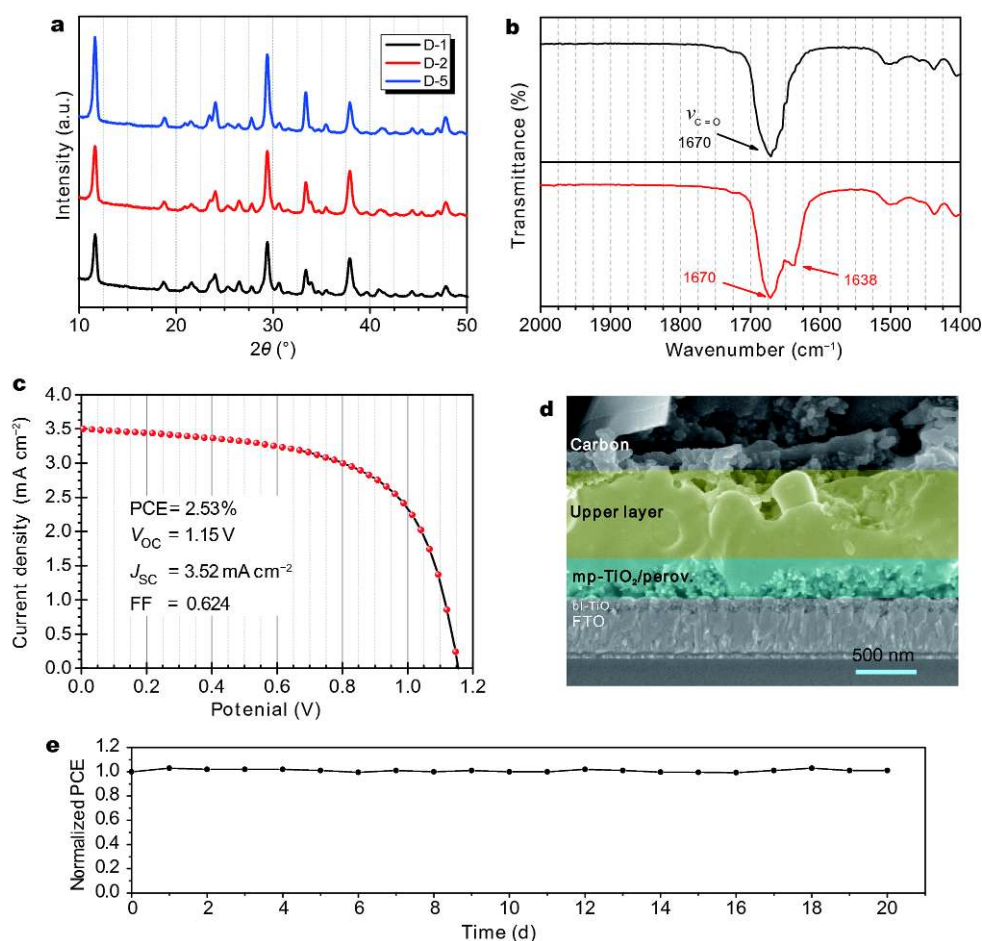


Figure 3 (a) XRD patterns of D-1, D-2, D-5 films; (b) FT-IR of T-1 solution (black line) and T-2 solution (red line); (c) champion cell's J - V characteristics of the PSC devices based on D-40 films; (d) cross-sectional SEM image of a full cell based on D-40 films, the scale bar is 500 nm; (e) normalized PCE of the device based on D-40 films.

Table 1 Typical device performance of PSCs

	J_{sc} (mA cm^{-2})	V_{oc} (V)	PCE (%)	FF
D-10	–	–	–	–
D-20	1.89	0.83	0.82	0.52
D-40	3.25	1.10	2.18	0.61

method to fabricate fully-inorganic PSCs using carbon as hole-transporting material and photocathode. A material and phase evolution process from tetragonal CsPb_2Br_5 to quasi-cubic CsPbBr_3 was found and characterized. SEM, XRD patterns, UV-vis spectra and FT-IR spectra were used to investigate the evolution process. We proposed a hypothesis to explain the evolution process. The best cell based on quasi-cubic CsPbBr_3 films presented a PCE of 2.53% with long stability over 20 d under ambient air conditions without any encapsulation. Our results are helpful to the development of CsPbBr_3 based PSCs. In

addition, the facile way to synthesize CsPb_2Br_5 could enlighten other researchers to develop and design more applications of it.

Received 7 July 2017; accepted 12 September 2017;
published online 27 October 2017

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Acknowledgements This work was supported by the National Basic Research Program of China (2016YFA0202400 and 2015CB932200), the National Natural Science Foundation of China (21403247), Distinguished Youth Foundation of Anhui Province (1708085J09), the Fundamental Research Funds for the Central Universities (2017XS079) and the Major/Innovative Program of Development Foundation of Hefei Center for Physical Science and Technology (2016FXZY003)

Author contributions Ding X conducted the main experiments and wrote this manuscript. Zhu J directly guided this research including the designing, modifying and optimizing work related to this manuscript. Dai S supervised the projects and carefully reviewed and modified this manuscript. Ren Y, Wu Y, Xu Y, Tasawar H, Ahmed A, Li Z, Huang Y provided help in the fabrication of devices and methods of characterization. All authors contributed to the general discussion about this work.

Conflict of interest The authors declare that they have no conflict of interest.

Supplementary information Supporting data are available in the online version of the paper.



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连续沉积法制备碳电极无机钙钛矿太阳电池

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摘要 有机无机杂化钙钛矿是一种有潜力的光伏材料. 然而, 对于环境的高度敏感性限制了它的实际应用. 因此我们需要发展全无机钙钛矿材料. 本文通过连续沉积法制备了效率达到2.53%的碳电极CsPbBr₃太阳电池, 并且它能够在无封装的情况下稳定20天. 在制备CsPbBr₃薄膜的过程中, 我们首次发现从CsPb₂Br₅到CsPb₂Br₅-CsPbBr₃混合物到CsPbBr₃的转变. 该研究结果对于无机钙钛矿太阳电池的发展和CsPb₂Br₅基光电器件具有帮助作用.