Defect Passivation of CsPbBr$_3$ with AgBr for High-Performance All-Inorganic Perovskite Solar Cells

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CsPbBr$_3$-based perovskite solar cells possess both high humidity and temperature tolerance, and also have excellent long-term stability in air, but the defect states in the bulk perovskite are always an obstacle for further improvement of their efficiency. Herein, a doping strategy is demonstrated in which AgBr is directly added to the precursor solution in the first step. An appropriate amount of Ag$^+$ ion doping can passivate the defects in bulk or at grain boundaries, reduce the defect density of the film, and form a pinhole-free film. Finally, the best power conversion efficiency of the solar cell reaches to 6.92% and exhibits a long-term stability in a humid environment. This work provides a simple and effective method for improving the quality of CsPbBr$_3$ film and the performance of CsPbBr$_3$-based perovskite solar cells.

1. Introduction

Within a few years of development, the performance of perovskite solar cells (PSCs) has exceeded the highest efficiency of the solar cells based on semiconductor compounds such as CdTe and CIGS. Although the efficiency of perovskite solar cells based on organic-inorganic hybrid perovskites has reached 23.5% from initial 3.8%, these materials are easily decomposed under high temperature or moisture. In contrast, all-inorganic perovskites based on Cs cation (CsBX$_3$, B = Pb, Sn; X = Cl, Br, I) have good thermal stability, among which CsPbBr$_3$ has an excellent both moisture stability and thermal stability. Compared with the lower decomposition temperature of MAPbI$_3$ (220°C), the degradation temperature of CsPbBr$_3$ is as high as 580°C. Moreover, MAPbBr$_3$ is more easily decomposed in a humid environment because MA$^+$ has a higher hydrophilicity.

Despite excellent stability, CsPbBr$_3$ has a lower defect formation energy than iodine-rich CsPbI$_{3-x}$Br$_x$ (x < 1) perovskite, and so CsPbBr$_3$ films are more likely to have higher defect concentration. To address this problem, a certain proportion of I$^-$ can be added into the CsPbBr$_3$ perovskite, but the phase transition temperature can be higher as I$^-$ proportion increases. For example, the phase transition temperature of the cubic CsPbI$_3$ can reach up to 310°C while changing to the orthogonal phase occurs at room temperature, so iodine-rich perovskite has lower resistance to air environment. Therefore, reducing defects in CsPbBr$_3$ film can make it competitive in photovoltaic research and industry.

As we all know, the surface defects and pinholes of the perovskite film are important factors that restrict the device efficiency. In other words, the performance of the device can be effectively improved by surface passivation and extending the carrier lifetime. Recently, many studies have focused to improve the quality of perovskite film through compositional engineering. For example, alkali metal cations and lanthanide ions doping can passivate interface defects and reduce nonradiative recombination; organic short-chain ligand passivation can enhance the charge transfer rate; and so on. It is worth noting that the B-site doping with a smaller ionic radius (compared with Pb ions) can make the tolerance factor closer to 1, thereby forming a perfect cubic lattice structure. Thus, B-site doping is a promising compositional engineering strategy to improve the quality of perovskite films.

In this article, we demonstrate a silver halide doping strategy to reduce the defect density of CsPbBr$_3$ and improve the film quality. The analysis of the optical characterization shows that the introduction of appropriate amount of AgBr can significantly...
increase the photoluminescence (PL) intensity, indicating that the defect density in CsPbBr$_3$ film is significantly reduced, which is consistent with the trap density calculated by using space-charge-limited current (SCLC) measurement. Furthermore, the time-resolved PL (TRPL) curve shows that doping with low concentration AgBr (0.01 M) can extend the carrier lifetime and reduce the nonradiative recombination. By observing the thin-film surface of top scanning electron microscopy (SEM) images before and after AgBr doping, it was found that a proper amount of AgBr doping can make the film surface pinholes-free.

2. Results and Discussion

First, we studied the X-ray diffraction (XRD) patterns of pure phase and doped with different concentrations of Ag$^+$ ions (see Figure 1a). Obviously, three main peaks (100), (110), and (111) at around 15.15°, 21.51°, and 26.42° are detected in all sorts of films without any peak shifts, indicating that the introduction of Ag$^+$ ions does not change the crystal lattice spacing.$^{[19]}$ In addition, the intensity of the (110) peak increases slightly and fewer impurity peaks can be observed when doped with 1% Ag$^+$ ions, indicating that appropriate amount of Ag$^+$ doping can improve the crystallization and quality of the film. In addition, the radius of Ag$^+$ ions (1.15 Å) is very similar to that of Pb$^{2+}$ ions (1.19 Å), so we believe that some Ag$^+$ ions replace the Pb site without changing the lattice spacing. To further verify the interaction between Ag$^+$ ions and perovskite, we used the X-ray photoelectron spectroscopy (XPS) measurement to collect the detection signals of different orbital peaks, including Cs 3d, Pb 4f, Br 3d, and Ag 3d. As shown in Figure 1b–d, it can be observed that Cs 3d and Pb 4f orbital peaks have no shift after Ag$^+$ ion doping. However, the Br 3d orbital peak appeared more obvious shift, while the Pb 4f orbital peak hardly moves, indicating that Ag$^+$ ion does not interact with Cs$^+$ ion and Pb$^{2+}$ but interacts with halide and thus redistributed electron density caused by the different binding interaction between Ag$^+$ and Br$^-$.[$^{[20,21]}$]

In detail, a stronger interaction would be formed between Ag$^+$ and Br compared with Pb–Br bond, when the more electronegative Ag$^+$ ion replaces the Pb$^{2+}$ ion, which is consistent well with the conclusion that the Ag$^+$ ion can replace the Pb position explained by XRD measurement. In addition to replacing the Pb position, Ag$^+$ ions may also form strong coordination bonds with halide ions at the grain boundaries to play a passivation effect.$^{[22,23]}$ This part would be mentioned again later. Furthermore, we also detected the Ag orbital peak during the XPS test (see Figure 1e), which further proves that Ag$^+$ ions are doped into the perovskite film. Finally, as shown in Figure S1, Supporting Information, the energy dispersive spectroscopy (EDS) mapping image shows that each element are doped into the perovskite film. Finally, as shown in Figure 1f, we show a schematic diagram of the perovskite host lattice after doping Ag$^+$ ions.

Subsequently, we studied the effect of different AgBr doping levels on optical properties. Figure 2a shows the relative PL spectra of AgBr doped with different concentrations. It was found that the PL intensity can be significantly improved after being doped with 1% AgBr compared with the pure CsPbBr$_3$ film, indicating

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**Figure 1.** a) XRD patterns of perovskite films with different Ag$^+$ ion doping concentrations (0, 0.01 M, 0.02 M) and XPS spectra of CsPb$_{0.99}$Ag$_{0.01}$Br$_3$ films at the b) Cs 3d, c) Pb 4f, and d) Br 3d regions. e) Ag 3d$_{5/2}$ and Ag 3d$_{3/2}$ orbital peaks also detected in CsPb$_{0.99}$Ag$_{0.01}$Br$_3$ film. f) The host perovskite lattice illustration after Ag$^+$ ion doping. The octahedron in which Pb is replaced with Ag is shown in lighter color.
that doping with appropriate levels of AgBr can reduce the defect density of the film, while the PL intensity is significantly weakened when the doping level of AgBr increased to 2%, indicating that excessive doping would increase the defect density, thus deteriorating film quality. In detail, they may react with Br to form AgBr and thereby destroy the CsPbBr\textsubscript{3} perovskite. In addition, we conducted the UV–vis absorbance spectra and converted corresponding (dah), versus energy (h\nu) curves (see Figure S2, Supporting Information) and found that the absorption intensity did not change significantly after 1% doping. However, when the doping ratio increased to 2%, the absorption intensity decreased significantly. Meanwhile, there are almost no change in the perovskite bandgaps before and after doping. Furthermore, we tested the corresponding TRPL curve at the optimal emission wavelength (536 nm) (see Figure 2b). Then we used the biexponential function formula for fitting, and also calculated the average life (\tau_\text{ave}) of different films. The average carrier lifetime (\tau_\text{ave}) under 1% AgBr doping increases from the initial 1.54 to 1.84 ns, indicating that the introduction of a suitable amount of AgBr can extend the carrier lifetime and inhibit nonradiative recombination in perovskite films. However, the carrier lifetime decreases significantly to 0.92 ns when the AgBr doping ratio increases to 2%, indicating that excessive AgBr would introduce more defects and increase the nonradiative recombination path, which is obviously detrimental to the film quality. The relevant TRPL parameters are shown in Table S1, Supporting Information, where \tau_1 and \tau_2 are the short-life component of carrier nonradiative recombination and the long-life component of effective carrier recombination, respectively. The lifetime and specific gravity of \tau_2 reach the maximum at 1% Ag\textsuperscript{+} ions doping ratio, while the average lifetime reaches up to 1.84 ns. However, with the further increase in the doping concentration (0.02 M AgBr), the gravity of \tau_2 decreases sharply to 15.08%, while the gravity of \tau_1 quickly increases to 84.92%, and the average lifetime \tau_\text{ave} reaches the minimum value of 0.92 ns. It was further confirmed that excess Ag\textsuperscript{+} ions would introduce more defect sites and increase nonradiative recombination paths, thereby reducing the average carrier lifetime. Kang et al. showed that the defect formation energy of bromine-rich perovskite is much lower than that of iodine containing inorganic perovskite, and the dominant energy-level defects are concentrated on the surface by the first-principles calculations. Therefore, passivation film surface would be able to effectively improve the quality of CsPbBr\textsubscript{3} films and the photovoltaic performance of the devices. Furthermore, we used the device structure (FTO/perovskite/carbon) shown in Figure 2c to test the SCLC published under dark conditions (see Figure 2d,e). The value of \eta_\text{trap} is calculated by the formula: \eta_\text{trap} = 2e\epsilon_0V_{\text{TFL}}/qL^2, where \epsilon_0 is the vacuum permittivity (8.85 × 10\textsuperscript{-12} F m\textsuperscript{-1}), \nu is the dielectric constant of CsPbBr\textsubscript{3} (≈22),\textsuperscript{130} q is the electron charge (1.6 × 10\textsuperscript{-19} C), L represents the thickness of the perovskite film (≈400 nm), and V_{\text{TFL}} is trap filled limit voltage (0.333, 0.231, and 0.482 V for the CsPbBr\textsubscript{3}, CsPb\textsubscript{0.99}Ag\textsubscript{0.01}Br\textsubscript{3}, and CsPb\textsubscript{0.98}Ag\textsubscript{0.02}Br\textsubscript{3}, respectively).

We observed that \eta_\text{trap} rapidly decreased from 5.059 × 10\textsuperscript{15} cm\textsuperscript{-3} in CsPbBr\textsubscript{3} film to 3.509 × 10\textsuperscript{15} cm\textsuperscript{-3} (CsPb\textsubscript{0.99}Ag\textsubscript{0.01}Br\textsubscript{3} film) and then increased to 7.323 × 10\textsuperscript{15} cm\textsuperscript{-3} (CsPb\textsubscript{0.98}Ag\textsubscript{0.02}Br\textsubscript{3} film). It can be clearly seen that the appropriate amount of Ag\textsuperscript{+} doping significantly reduces the defects of the perovskite film, but the excessive Ag\textsuperscript{+} ion doping introduces more defect states in the film.

Figure 2. a) PL and b) TRPL spectra for different CsPb\textsubscript{x−y}Ag\textsubscript{y}Br\textsubscript{3} (x = 0–0.02) films. c) Dark J–V measurement device structure: FTO/perovskite/carbon. d–f) Dark J–V curves characterization in different perovskite films.
To study the effect of the doping on the thin films, we used SEM characterization to observe the surface morphology of CsPb$_{1-x}$Ag$_x$Br$_3$ ($x = 0, 0.01, 0.02$) thin films. As shown in Figure 3, several pinholes on the surface of the undoped film can be observed, while the numbers of pinholes are significantly reduced after 1% AgBr doping. In contrast, excessive Ag$^+$ ions doping brings more pinholes, which is obviously detrimental to the surface quality of the film. The improved film coverage helps to improve the photovoltaic performance of devices, especially the further improvement of fill factor (FF). As shown in Table S2, Supporting Information, the FF of CsPbBr$_3$ solar cells doped with 1% AgBr has a significant improvement from 78.53% to 83.95% compared with the pure phase devices.

Finally, we made CsPbBr$_3$, 1% Ag-doped, and 2% Ag-doped CsPbBr$_3$ thin films into devices with a structure of FTO/$c$-TiO$_2$/m-TiO$_2$/perovskite/carbon (see Figure 4a), which avoids the usage of traditional expensive organic hole transport layer (HTL). The SEM cross-sectional image of a carbon-based solar device without HTL is shown in Figure 4b, where the thickness of the FTO layer is $\approx 650$ nm, the $c/m$-TiO$_2$ with thickness of $\approx 390$ nm acts as the electron transport layer, the perovskite layer with the thickness of $\approx 400$ nm performs as a light-absorbing layer, and the carbon electrode at the top acts as an anode. The photovoltaic parameters and $J$–$V$ curves measured under AM1.5G illumination are shown in Table S2, Supporting Information and Figure 4c,d. The efficiency of the solar cell based on pure phase CsPbBr$_3$ is only 6.31%, but the efficiency rises to the maximum value of 6.92% when the doping ratio of AgBr increased to 1%. Compared with pure phase perovskite, the open-circuit voltage ($V_{oc}$), short-circuit current ($J_{sc}$), and FF increase to different degrees, which are 1.13 V, 7.87 mA cm$^{-2}$, and 80.07%, respectively. Specifically, the improvement of $V_{oc}$ and $J_{sc}$ can be attributed to the following: 1) a reduction in the density of defect states of the CsPb$_{0.99}$Ag$_{0.01}$Br$_3$ film can effectively reduce the nonradiative recombination path; 2) the surface and grain boundaries of the film can be passivated and the carrier life can be extended, which is consistent with the aforementioned conclusion that Ag$^+$ ions form coordination with halide at the surface and grain boundaries. However, the solar cell efficiency drops rapidly to 5.30% when the AgBr doping ratio increases to 2%, indicating that excessive AgBr adversely affects the photovoltaic performance of the devices. Moreover, we show the box diagram of each photovoltaic parameter before and after 1% Ag$^+$ ion doping (see Figure 4e–h). Compared with the undoped one, the box body of efficiency exhibits a higher and narrower distribution after doping with Ag$^+$ ion, which indicates that the introduction of an appropriate amount of Ag$^+$ ions not only enhances the photovoltaic parameters but also improves the repeatability. In addition, we tested the long-term stability of CsPbBr$_3$ and CsPb$_{0.99}$Ag$_{0.01}$Br$_3$ devices under 80% relative humidity (see Figure S3, Supporting Information). Notably, the efficiency of CsPbBr$_3$-based solar cell...
drops to 0.5 of the initial value after being stored for ≈600 h, while the CsPb$_{0.09}$Ag$_{0.01}$Br$_3$-based solar cell still maintains more than 80% of initial efficiency after 1000 h. It shows that the introduction of appropriate amount of Ag$^+$ can effectively improve PSCs conversion efficiency.

3. Conclusion

In summary, we proposed an element doping strategy and successfully applied it to the two-step immersion method. Ag$^+$ ions replace the Pb position and form a strong coordination bond with Br$^-$ ions. Furthermore, we believe that some Ag$^+$ ions are concentrated near the surface or grain boundaries and coordinated with halides to play a passivation effect. A series of optical characteristics show that the introduction of an appropriate amount of Ag$^+$ ions effectively reduces the defect density of the films, which is also quantitatively verified with the trap-filled limit voltage ($V_{TFL}$) obtained by the SCLC. In addition, the SEM surface image also confirms that a large number of pinholes are eliminated and finally a pinhole-free film surface is formed when introducing 1% Ag$^+$ ions. Finally, the efficiency of the CsPb$_{0.09}$Ag$_{0.01}$Br$_3$-based device increases from 6.31% to 6.92%, and also shows an excellent long-term stability compared with CsPbBr$_3$-based device, indicating that Ag$^+$ ion doping effectively passivates perovskite surface defects. In the future, we will consider applying 2D materials to the perovskite light absorption layer to achieve high moisture stability and broad depletion region. [34–36]

4. Experimental Section

Materials: Lead bromide (PbBr$_2$, 99.0%), cesium bromide (CsBr, 99.999%), and silver bromide (AgBr, 99.9%) were purchased from Aladdin Industrial Corporation. N,N-Dimethylformamide (DMF, 99.8%, anhydrous) was purchased from Sigma-Aldrich. All chemicals were used without further purification.

Device Fabrication: FTO glass (8 $\Omega$ square$^{-1}$) was separately washed with deionized water, acetone, and absolute ethanol for 15 min, and then treated with ultraviolet ozone for 15 min. First, an titanium oxide solution was spin-coated on FTO glass substrate at 3000 rpm for 30 s, and then annealed in air at 500°C for 30 min to form compact TiO$_2$ (c-TiO$_2$) layer. Titanium oxide paste (Greatcell Solar, 30 NR-D with an average particle size of 30 nm) was spin-coated on c-TiO$_2$ layer at 4000 rpm for 10 s and annealed in air at 500°C for 30 min to obtain the mesoscopic TiO$_2$ (m-TiO$_2$) film. Subsequently, AgBr and PbBr$_2$ powders were added into DMF solvent at certain ratio to form a PbBr$_2$@AgBr mixed solution. The mixed solution was then deposited on the m-TiO$_2$ film at 2000 rpm for 30 s, followed by heating on a hot plate at 90°C for 30 min. The PbBr$_2$@AgBr film was then immersed in CsBr solution (0.07 M) at 50°C for 10 min and then heated at 250°C on hot plate for 5 min to form a bright yellow CsPb$_x$Ag$_{1-x}$Br$_3$ ($x = 0–0.02$) films. And finally, the carbon paste was uniformly coated on the perovskite film by using a doctor-blade method and heated at 120°C for 10 min.

Characterization: The surface morphology and EDS mapping of the perovskite film were characterized by a field emission scanning microscope (GeminiSEM 300). The crystal structure of the films surface was recorded by X-ray diffractometer (DR8 Advance) with Cu Kα ($\lambda = 1.5406$ Å) radiation. The XPS characterization was performed using the Thermo Scientific K-Alpha instrument with Al Kα radiation ($h\nu = 1486.6$ eV). PL and TRPL spectra were detected by an Edinburgh Instruments FLS980 steady-state/transient fluorescence spectrometer. UV–vis absorption spectra of different perovskite films were collected using Cary 5000 instrument. The trap state density was measured with a SCLC model using the Mott–Gurney law based on FTO/perovskite/carbon recorded from 1 to 5 V. The J–V curve of the solar cell was measured by a photovoltaic simulator (Sirius-SS150A-D) under AM1.5G simulated solar illumination. The active area is 0.09 cm$^2$ performed by a black metal mask.

Supporting Information

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

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

Research data are not shared.

Keywords

defect passivation, doping, grain boundary passivation, perovskite solar cells

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