

Article

Optimization of Tin Fluoride Additive Concentration for High-Performance Sn–Pb Perovskite Solar Cells

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Abstract

Tin–lead halide perovskite is a promising narrow-bandgap absorber for high-performance perovskite solar cells. However, the easy oxidation of Sn^{2+} and the resulting defect formation still limit these films' quality and photovoltaic performance. Tin fluoride (SnF_2) is widely used as an antioxidant additive in Sn-containing perovskites, but its optimal concentration remains strongly dependent on the specific perovskite composition. Herein, we systematically investigate the influence of SnF_2 concentration on the film quality and device performance of methylammonium-free Sn–Pb perovskite solar cells. By varying the SnF_2 content from 0 to 15% relative to SnI_2 , we find that an appropriate amount of SnF_2 can effectively improve the surface morphology, enhance crystallinity, promote preferred crystal orientation, and suppress defect-assisted non-radiative recombination. In particular, the film with 10% SnF_2 exhibits the smoothest surface, with a reduced root-mean-square roughness, enhanced photoluminescence intensity, and a lower trap density compared with the control films. As a result, the optimized device delivers a champion power conversion efficiency of 19.15%, significantly outperforming the control device. This work demonstrates the importance of SnF_2 concentration optimization and provides a useful guideline for improving MA-free Sn–Pb perovskite solar cells.

Keywords: Sn–Pb perovskite solar cells; tin fluoride additive; concentration optimization

1. Introduction

Mixed tin–lead halide perovskite solar cells (Sn–Pb PSCs) have attracted extensive attention in recent years due to the excellent optoelectronic properties of mixed Sn–Pb perovskites, such as a high absorption coefficient, tunable narrow bandgap, high carrier mobility, and reduced Pb content [1–3]. However, the photovoltaic performance of Sn–Pb PSCs is closely related to the chemical stability of Sn^{2+} . To be more specific, Sn^{2+} can be readily oxidized to Sn^{4+} , which induces Sn vacancies, p-type self-doping, and serious non-radiative recombination [4,5]. Therefore, it is vital to suppress Sn^{2+} oxidation and regulate the crystallization process to form high-quality Sn–Pb perovskite films with compact morphology, high crystallinity, and low trap density.

Since SnF_2 was first introduced into CsSnI_3 perovskite solar cells by Mathews et al., resulting in a power conversion efficiency (PCE) of 2.02%, it has become one of the most commonly used additives in pure Sn-based and mixed Sn–Pb perovskites [6–8]. The addition of SnF_2 can provide a Sn-rich environment, reduce Sn-vacancy formation, suppress



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trap-induced non-radiative recombination, and improve film quality. Nevertheless, there is still no clear consensus on the optimal SnF_2 concentration. Different studies have reported different preferred amounts, ranging from relatively low contents such as 5% (relative to SnI_2) to commonly used values around 10%, and even higher levels such as 15%, depending on the perovskite composition [9–12]. Considering the relatively poor thermal stability of methylammonium (MA) organic cations, an MA-free Sn–Pb perovskite composition is expected to exhibit improved thermal robustness under continuous light illumination [13,14].

In this work, we systematically investigate the influence of SnF_2 concentration on the quality of MA-free Sn–Pb perovskite films and the performance of corresponding PSCs. By meticulously studying the crystallization morphology, optical properties, defect characteristics, and photovoltaic performance of Sn–Pb perovskite films with different SnF_2 concentrations, the optimal amount of 10% SnF_2 additive is determined. Finally, with the employment of 10% SnF_2 concentration, the optimized Sn–Pb PSCs achieve a power conversion efficiency (PCE) of 19.15%.

2. Experimental Section

2.1. Materials

FTO glass substrates (sheet resistance = $15 \Omega \text{ sq}^{-1}$, thickness = 1.6 mm), lead iodide (PbI_2 , 99.99%), tin iodide (SnI_2 , 99.99%), and bathocuproine (BCP, 99.99%) were purchased from Advanced Election Technology. Formamidinium iodide (FAI, 99.5%) was purchased from Greatcell solar Materials Pty Ltd. Cesium iodide (CsI , 99.9%) was purchased from Shanghai Macklin Biochemical Technology Co., Ltd, Shanghai, China. Phenyl-C61-butyric acid methyl ester (PCBM, 99%) was purchased from Xi'an Polymer Light Technology in China. Ethylenediammonium diiodide (EDAI_2 , >98.0%) was purchased from Tokyo Chemical Industry Co., Ltd, Tokyo, Japan. Chlorobenzene (99.99%, anhydrous), N,N-dimethylformamide (DMF, >99.9%, anhydrous), dimethyl sulfoxide (DMSO, >99.8%, anhydrous) and tin(II) fluoride (SnF_2 , 99%) were purchased from Sigma-Aldrich, USA. Poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate) (PEDOT:PSS) aqueous solution (Clevious PVP AI 4083) was purchased from J&K Scientific. All chemicals were used without further purification.

2.2. Preparation of the Perovskite Precursors

A 1.8 M $\text{FA}_{0.85}\text{Cs}_{0.15}\text{Pb}_{0.5}\text{Sn}_{0.5}\text{I}_3$ mixed perovskite precursor solution was prepared by dissolving PbI_2 (414.99 mg), SnI_2 (335.3 mg), FAI (263.1 mg), and CsI (70.1 mg) in 1 mL anhydrous DMF/DMSO mixture solvent (3/1, *v/v*). The precursor solution was stirred overnight and filtered by 0.22 μm PTFE filters before use.

2.3. Device Fabrication

FTO glass substrates were cleaned with deionized water, acetone, ethanol, and isopropyl alcohol for 15 min at each step. After being dried by dry nitrogen, the FTO was treated with ultraviolet ozone (UV-O_3) for 15 min. PEDOT:PSS was spun on the FTO substrate at 4000 rpm for 30 s, followed by an annealing process of 150 $^\circ\text{C}$ for 20 min. The films were then quickly transferred to a N_2 glove box. Subsequently, the perovskite precursor solution with different SnF_2 concentrations was deposited on the PEDOT:PSS layer through spin-coating in a two-step program at 1000 and 4000 rpm for 10 and 40 s, with accelerations of 200 rpm s^{-1} and 1000 rpm s^{-1} , respectively. After 20 s in the second step, 300 μL of chlorobenzene was dropped on the spinning substrate. The prepared films were then annealed at 100 $^\circ\text{C}$ for 10 min. Subsequently, the 120 μL 0.5 mg/mL EDAI_2 IPA solution was dropped on perovskite layers with 4000 rpm for 20 s and annealed 100 $^\circ\text{C}$

for 5 min. Then, PCBM (20 mg/mL in CB) was spin-coated at 1200 rpm for 35 s without annealing. BCP (0.5 mg/mL in IPA) was then dynamically spin-coated at 4000 rpm for 30 s. Finally, Ag was sequentially deposited by thermal evaporation at low pressure ($<10^{-4}$ Torr). The active area defined by a mask aperture was calibrated to be 0.067 cm^2 . All solutions were filtered through the $0.22\text{ }\mu\text{m}$ PTFE filter before use, except PEDOT:PSS, which used $0.45\text{ }\mu\text{m}$ PES filters.

2.4. Characterizations

XRD patterns were collected on a Rigaku SmartLab using Cu-K α radiation. UV-vis spectra of the perovskite films were measured on a Shimadzu UV-3600 UV-vis-NIR spectrophotometer. SEM images were observed by a field-emission scanning electron microscope (Thermo scientific Apreo C, USA). PL was employed by a micro-Raman spectrometer (Renishaw in Via) with an excitation laser wavelength of 532 nm. J - V characteristics were measured with a Keithley 2450 source meter under one-sun illumination (AM 1.5 G, 100 mW cm^{-2}) using a solar simulator (Newport LCS-100) equipped with a 100 W Xenon ARC lamp (Newport 6252), in which light intensity was calibrated by an NREL-calibrated Si solar cell equipped with a KG-2 filter. SCLC and dark J - V characteristics were measured with a Keithley 2450 source meter in darkness.

3. Results and Discussion

3.1. Improved Film Quality

Systematically clarify the influence of different SnF_2 concentrations on Sn-Pb perovskite film formation. As shown in Figure 1, PEDOT:PSS films are prepared on UV- O_3 -treated FTO substrates. Subsequently, the perovskite precursor solution with varying contents (0%, 5%, 10%, and 15% relative to mol percentage of SnI_2) is deposited on the PEDOT:PSS films with chlorobenzene used as an antisolvent during film formation. After $100\text{ }^\circ\text{C}$ annealing for 10 min, Sn-Pb perovskite thin films are obtained for systematic characterization and device fabrication. The detailed parameters and fabrication process are provided in Section 2.

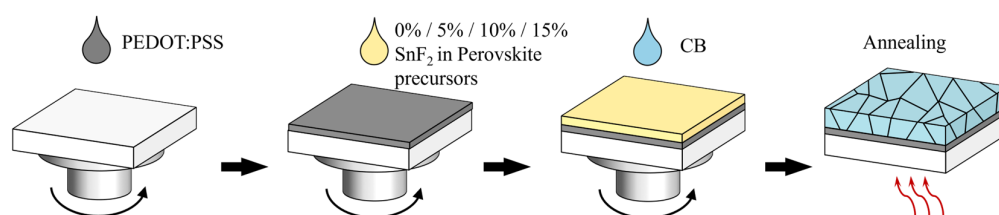


Figure 1. Schematic illustration of the fabrication process of Sn-Pb perovskite films.

Atomic force microscopy (AFM) was carried out to study the effect of varying SnF_2 additions on Sn-Pb perovskite films' surface roughness. A lower root-mean-square roughness (R_q) is favorable for improving interfacial contact and suppressing interfacial recombination. As shown in Figure 2a, the control film without SnF_2 exhibits a relatively rough surface with an R_q of 36.1 nm, indicating an inhomogeneous film surface. After introducing SnF_2 , the surface morphology of the Sn-Pb perovskite films is gradually improved. The R_q value decreases from 36.1 nm for the control film to 31.3 nm and 27.4 nm for the films with 5% and 10% SnF_2 , respectively. However, further increasing the SnF_2 concentration to 15% leads to an increased R_q value of 33.6 nm. This result can be attributed to an appropriate amount of SnF_2 suppressing Sn^{4+} -induced defect formation, thereby improving the surface uniformity of Sn-Pb perovskite films, but excesses of 15% SnF_2 cause the accumulation of residual SnF_2 or F-rich species, resulting in the R_q value increasing [11]. Scanning electron microscopy (SEM) images further reveal that the incorporation of SnF_2

addition slightly increases grain size and effectively suppresses defect formation, with less “white phase” formation associated with Sn^{2+} oxidation-induced $[\text{SnI}_x]^{2-x}$ adduct formation (Figure S1, Supplementary Materials) [15,16]. In particular, the film with 10% SnF_2 presented the most compact morphology and the lowest “white phase”, which is consistent with the AFM results. However, it seems that SnF_2 unable to totally suppress Sn^{2+} oxidation indicating the limited antioxidant capability of a single SnF_2 additive. To further quantify the Sn^{2+} oxidation degree, X-ray photoelectron spectroscopy (XPS) was performed. Evidence shows that SnF_2 addition significantly suppresses Sn^{4+} formation, with a reduced peak area ratio of $\text{Sn}^{4+}/\text{Sn}^{2+}$ from 1.85 to 0.33 after SnF_2 treatment.

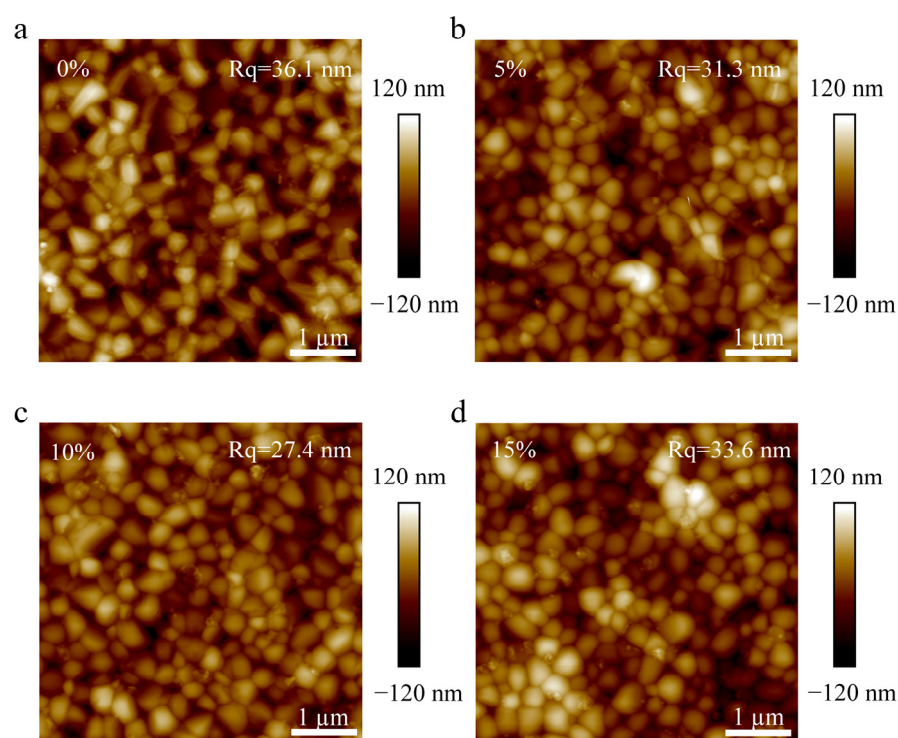


Figure 2. AFM images of Sn–Pb perovskite films with different SnF_2 concentrations: (a) 0%, (b) 5%, (c) 10%, and (d) 15% relative to SnI_2 .

To investigate the influence of varying SnF_2 amounts on the crystal structure and crystallinity of Sn–Pb perovskite films, X-ray diffraction (XRD) was carried out. As illustrated in Figure 3a, all films show identical diffraction peak positions, confirming that SnF_2 does not significantly alter the main crystal structure. However, the diffraction intensities are visibly enhanced after the introduction of SnF_2 , indicating the improved crystallinity. Particularly, the film with 10% SnF_2 showed the strongest diffraction peaks of (100) and (200), which is beneficial for carrier transfer and device efficiency according to previous reports [17–19]. This result is supported by the increased intensity ratio of the (100) diffraction peak for SnF_2 -treated films with respect to the control ($I_{(100)\text{-treated}}/I_{(100)\text{-control}}$), which exceeds 1 for all treated samples (Figure S2, Supplementary Materials). Moreover, the diffraction peak intensity ratio of (100)/(110) is further calculated, as shown in Figure 3b. The (100)/(110) ratios of all SnF_2 -treated films are higher than that of the control film, with the 10% SnF_2 film showing the highest value. However, the highest relative intensities of (111)/(200) peaks are observed at 5% SnF_2 , suggesting different SnF_2 concentrations preferentially promote different crystallographic orientations [20,21]. Therefore, these results indicate that an appropriate amount of SnF_2 can optimize the crystallinity and orientation of Sn–Pb perovskite films, particularly at a concentration of 10%.

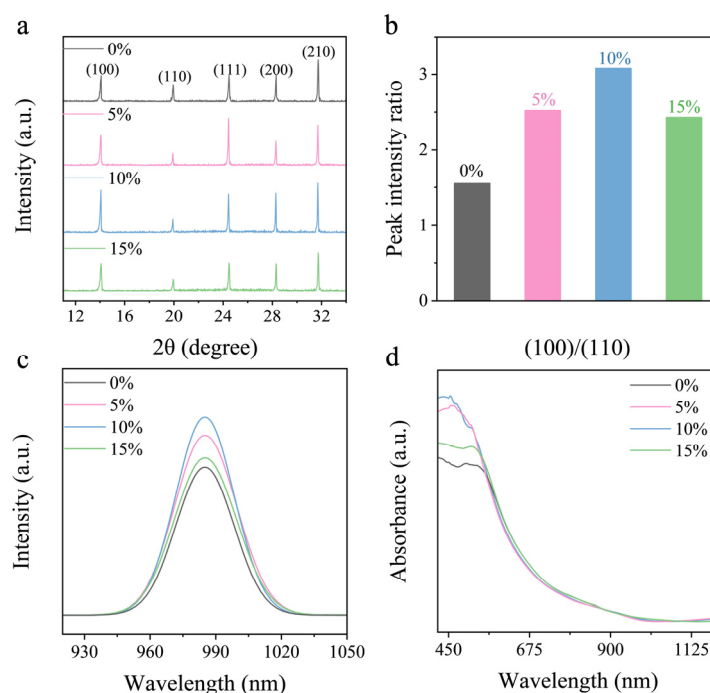


Figure 3. (a) XRD patterns, (b) corresponding peak intensity ratios of (100)/(110), (c) steady-state PL patterns, and (d) UV-vis-NIR absorbance spectra of Sn-Pb perovskite films with varying SnF₂ concentrations.

In Figure 3c, steady-state photoluminescence (PL) measurements were performed to evaluate the non-radiative recombination of Sn-Pb perovskite films. Sn-Pb perovskites with different SnF₂ concentrations deposited on the glass substrate were excited from the film side using a 532 nm laser. As a result, all films treated with SnF₂ exhibit enhanced PL intensities compared with the control film, suggesting suppressed defect-assisted non-radiative recombination after SnF₂ is introduced. Furthermore, the UV-vis-NIR absorbance spectra show a similar trend after the introduction of SnF₂, as shown in Figure 3d. The enhanced absorbance intensity of the SnF₂-treated films indicates improved film quality, which can be attributed to optimized crystallization and more compact film morphology. It is worth mentioning that the corresponding Tauc plots show an optical bandgap of approximately 1.26 eV, which agrees well with the PL emission peak located at ~985 nm. This result further confirms that SnF₂ incorporation does not change the bandgap and main crystal structure of Sn-Pb perovskite films (Figure S3, Supplementary Materials). The above results collectively confirm that the SnF₂ additive, especially at 10% concentration, can effectively suppress Sn²⁺ oxidation induced defect formation, thereby optimizing the films' morphology and crystallinity quality. Accordingly, the 10% SnF₂-treated film is selected as the "Target" sample for subsequent device studies, while the pristine film without SnF₂ is used as the "Control".

3.2. Enhanced Device Performance

To further evaluate the effect of SnF₂ additives on photovoltaic performance, as shown in Figure 4a, p-i-n Sn-Pb PSCs with a structure of FTO/PEDOT:PSS/FA_{0.85}Cs_{0.15}Pb_{0.5}Sn_{0.5}I₃/EDAI₂/PCBM/BCP/Ag were fabricated. As illustrated in Figure 4b, the control without SnF₂ treatment presents a PCE of 12.57% under forward scan, with a short-circuit current density (J_{SC}) of 29.72 mA/cm², an open-circuit voltage (V_{OC}) of 0.66 V and a fill factor (FF) of 63.60%. In contrast, the target device based on 10% SnF₂ treatment presents a much higher PCE of 19.15% under forward scan, with a J_{SC} of 30.07 mA/cm², a V_{OC} of 0.82 V and an FF of 77.57%. This performance is within the competitive values reported

for Sn–Pb PSCs, and notably, it is achieved using SnF₂ as the sole antioxidant additive (Table S2). The target device shows 52.34% enhancement in PCE compared with the control device, mainly owing to the significant improvement in V_{OC} and FF. This improvement can be attributed to the optimized film's quality, reduced defect density, and suppressed non-radiative recombination. Moreover, the SnF₂-modified devices, particularly the target group, exhibit improved reproducibility, with a narrower and more concentrated statistical distribution of photovoltaic parameters compared with the control, which is consistent with the J – V results (Figure 4c and Table S1, Supplementary Materials).

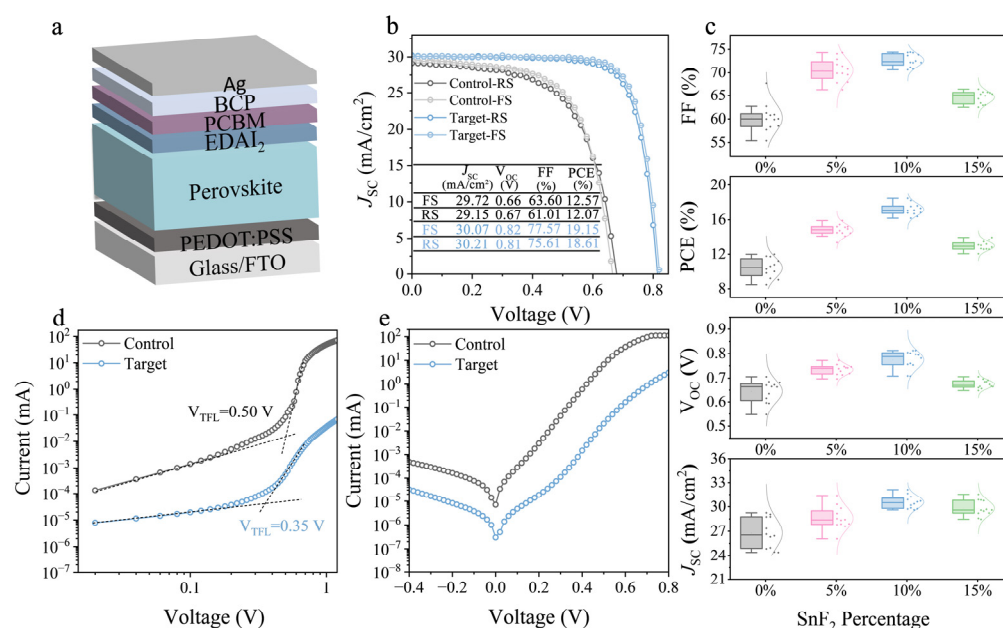


Figure 4. (a) Schematic illustration of the inverted Sn–Pb PSC architecture. (b) Champion J – V curves of the control and target devices. (c) Statistical distribution of the photovoltaic parameters of Sn–Pb PSCs with varying SnF₂ concentrations. (d) SCLC and (e) dark J – V measurements for the control and target devices.

To quantify the defect density in the Sn–Pb perovskite films, space-charge-limited current (SCLC) measurements were performed using the electron-only structure of FTO/SnO₂/Sn–Pb perovskite/PCBM/Ag. As shown in Figure 4d, the trap-filled limit voltage (V_{TFL}) decreases from 0.50 V for the control device to 0.35 V for the target device. The trap density (N_{trap}) is calculated by the following equation:

$$N_{trap} = \frac{2\epsilon\epsilon_0 V_{TFL}}{eL^2} \quad (1)$$

where ϵ , ϵ_0 , e , and L are the relative dielectric constant (~ 20), vacuum permittivity, elementary electric charge, and perovskite film thickness (~ 700 nm), respectively [22]. As a result, the target device exhibited a lower N_{trap} , of $1.58 \times 10^{15} \text{ cm}^{-3}$, than the control ($2.26 \times 10^{15} \text{ cm}^{-3}$) and other SnF₂ concentrations, indicating reduced vacancies and defects of the Sn–Pb PSCs (Figure 4d and Figure S4, Supplementary Materials) [23]. Additionally, the target device exhibits nearly an order of magnitude lower reverse saturation current than the control, suggesting that the leakage current and non-radiative recombination are effectively suppressed by SnF₂ modification, which is beneficial for improving the V_{OC} of the target device (Figure 4e). A similar decrease in the reverse saturation current is also observed in other SnF₂ concentrations (Figure S5, Supplementary Materials). Moreover, a smaller ideality factor is obtained after SnF₂ treatment, confirming that most carriers are collected prior to recombination (Figure S6, Supplementary Materials).

4. Conclusions

In summary, we systematically investigated the effect of SnF₂ concentrations on MA-free Sn–Pb perovskite films and the corresponding performance of devices. By meticulously studying the surface roughness, crystallization morphology, and optical properties of perovskite films, we found that the SnF₂ additive significantly improved the films' quality, with a smoother surface, improved crystallinity and fewer Sn²⁺ oxidation-related defects. Finally, the 10% SnF₂ additive concentration is determined. Under the optimized parameters, the target device delivers a champion PCE of 19.15%, with a J_{SC} of 30.07 mA/cm², an FF of 77.57% and a V_{OC} of 0.82 V, offering useful guidance for further improving the MA-free Sn–Pb PSCs' performance.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/coatings16070805/s1>, Figure S1: SEM images of Sn–Pb perovskite films with varying SnF₂ concentration; Figure S2. XPS spectra of Sn 3d of Sn–Pb perovskite films with varying SnF₂ treated; Figure S3: The intensity ratio of the (100) diffraction peak of the SnF₂-treated perovskite films relative to that of the control film; Figure S4. Tauc plots of Sn–Pb perovskite films with varying SnF₂ concentrations; Figure S5. SCLC curves of Sn–Pb PSCs containing 5% and 15% SnF₂ additives; Figure S6. Dark J – V curves of Sn–Pb PSCs containing 5% and 15% SnF₂ additives; Figure S7. Light-intensity-dependent V_{OC} measurements of Sn–Pb perovskite solar cells with varying SnF₂ treatment; Table S1: Comparison of photovoltaic parameters of MA-free Sn–Pb perovskite devices with representative reported works; Table S2. Statistical summary of photovoltaic parameters of 16 independent Sn–Pb PSCs with different SnF₂ additive concentrations [21,24–30].

Author Contributions: Y.L.: Data Curation, Writing—Original Draft; J.H.: Conceptualization, Methodology; Q.C.: Investigation, Formal Analysis; F.X.: Validation, Writing—Reviewing and Editing; X.Z.: Supervision, Project Administration. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Dataset available on request from the authors.

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