New insight into solvent engineering technology from evolution of intermediates via one-step spin-coating approach

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ABSTRACT The anti-polar solvent technique is an effective way to improve the film quality in a perovskite solar cell. In this work, we reveal the reason why chlorobenzene (CBZ) plays an important role in controlling the crystallization process. By investigating the formation of intermediate phases in the precursor solution, we observed that the $\text{CH}_3\text{NH}_3\text{I}$ (MAI)- PbI_2 -dimethylformamide (DMF) or MAI- PbI_2 -dimethylsulphoxide (DMSO) adducts have not yet formed until washed with non-polar solvent. The accelerated formation of intermediate phase yields high crystalline perovskite layers. Rapid solvent evaporation and retarded perovskite crystallization in one-step method are efficient to obtain high-quality perovskite films. Consequently, MAI- PbI_2 -DMSO intermediate shows neat rod-like structure with high crystallinity, which eventually transforms extremely dense and uniform perovskite films.

Keywords: perovskite solar cell, rapid-transformation, intermediate phases, anti-polar solvent technique

INTRODUCTION

During the past years, organic-inorganic hybrid perovskite cells have attracted much attention and revolutionized the prospects of photovoltaic technologies. The power conversion efficiency (PCE) of perovskite device has undergone a rapid development from 3.8% to 22.1% since the first attempt to introduce organic-inorganic hybrid perovskite materials as the light absorber [1–10]. Such rapid increase in performance can be ascribed to its excellent properties,

such as, ambipolar charge transport, high carrier mobility, long carrier diffusion, and large absorption coefficient [11,12]. Previous studies have uncovered that full surface coverage and low surface roughness are crucial for governing the device photovoltaic performance [5,6]. Therefore, to prepare high quality perovskite thin films, various methods have been developed, e.g., vacuum evaporation [6], vapor-assisted deposition [13–15], solvent engineering [16,17] and solution-based techniques [18–20]. Among these methods, solvent engineering technology has proven to be an efficient way for producing dense perovskite layers in a one-step deposition. This methodology can make use of non-polar solvent to regulate the perovskite crystallization process, and thus it has been widely used to produce uniform and dense perovskite films. For instance, Cheng and co-workers [17] reported that the conventional one-step spin-coating method followed by dropping chlorobenzene (CBZ) could lead to accelerated deposition of high-quality perovskite film. In contrast, Seok and co-workers found that the $\text{CH}_3\text{NH}_3\text{I}$ (MAI)- PbI_2 -dimethylsulphoxide (DMSO) intermediate phase retarded the reaction between MAI and PbI_2 [16]. Although the solvent engineering technology is a very useful experimental methodology, there remain two major challenges. First, the effect mechanism of the non-polar solvent on the precursor adducts which further governs the perovskite formation has not been systematically studied yet. Second, the reason why

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the MAI-PbI₂-DMSO adduct plays more important role in crystallization process than MAI-PbI₂-dimethylformamide (DMF) is unclear.

In this paper, we investigated the formation of MAI-PbI₂-DMF and MAI-PbI₂-DMSO intermediate phases by adding non-polar solvent (chlorobenzene) via a one-step coating method. The MAI-PbI₂-DMF and MAI-PbI₂-DMSO intermediate phases could not be found in the precursor solution until washed with CBZ. The results revealed that the non-polar solvent additive of CBZ improved the perovskite films in the following two aspects: (1) washed the excess solvent which is harmful for high-quality film formation; (2) promoted the formation of intermediate phases and thus retarded the crystallization of MAI and PbI₂. Moreover, through studying the adducts formation condition with different solvents, surface topography, crystal structure and crystallization kinetics, we found that DMSO constituted MAI-PbI₂-DMSO adduct showed neat rod-like structure and high X-ray diffraction peak intensity, leading to high-quality crystalline film. The perovskite solar cell annealed from MAI-PbI₂-DMSO adduct achieved a PCE up to 16.9%.

EXPERIMENTAL SECTION

Solar cell fabrications

Fluorine-doped tin oxide (FTO) glasses (Pilkington, TEC15) were etched with 0.8 mol L⁻¹ HCl aqueous and Zn powder. The etched glasses were cleaned with detergent, distilled water and ethanol, and sonicated for 30 min. By means of spray pyrolysis, the blocking TiO₂ layers (bl-TiO₂) were deposited on the as-prepared FTO which was followed by heating at 510°C for 30 min.

The mesoporous TiO₂ (mp-TiO₂) film was deposited on cooling bl-TiO₂ by spin-coating of the TiO₂ paste (Dyesol 30NR-T) following by calcining at 510°C for 20 min.

530 mg of PbI₂ and 183 mg of CH₃NH₃I were dissolved in 1 mL of solvent containing different ratios of DMSO and DMF (*v/v*), and then the solution was under magnetic stirring for 1 h. The completely dissolved solution was spin-coated onto the mp-TiO₂ layer at 1100 rpm for 10 s and 5000 rpm for 30 s. 0.75 mL of CBZ was quickly dripped onto the rotating substrate at 2 and 25 s after the onset of the second step (5000 rpm for 30 s) for S-0 and S-100 solution, respectively. The surface would turn turbid in absence of CBZ washing. The perovskite film was heated at 100°C for 20 min in order to obtain a dense film. Then, spiro-OMeTAD solution (25 μ L), which consisted 73 mg of spiro-OMeTAD, 28 μ L of 4-*tert*-butyl pyridine

and 17.5 μ L of lithium bis(trifluoromethanesulfonyl)imide (Li-TFSI) solution (520 mg of Li-TFSI in 1 mL of acetonitrile) in 1 mL of CBZ, was spin-coated on the perovskite film at 3000 rpm for 20 s. Finally, Au electrode with a thickness of 60 nm was deposited by using thermal evaporation under vacuum at a constant evaporation rate of 0.6 nm s⁻¹.

Characterization

The morphology of the perovskite film was studied by a field emission scanning electron microscope (FESEM, FEI Sirion 200, Netherland). The crystal phase was obtained with X-ray diffraction (XRD, X'Pert Pro, Netherland) using Cu K α beam (λ =1.54 Å). The photocurrent density-voltage (*J*-*V*) curves were measured under one sun illumination (AM 1.5G, 100 mW cm⁻²) with a solar simulator (94043A, USA) equipped with Keithley 2400 source meter. When measuring, a mask with 0.09 cm² aperture area was used to avoid light scattering through the sides and define the effective area of the device. Fourier transform infrared spectroscopy (FTIR, Thermo Fisher IS50R, USA) was used to collect the spectral data for various solution and intermediates in the range of 4000–600 cm⁻¹.

RESULTS AND DISCUSSION

In this work, to clarify the solvents containing DMF and DMSO with different ratios, we named S-0, S-15, S-30, S-70, S-85, and S-100 solution in the following discussions. These numbers represent the percentage of DMSO in the mixed solvent. The P-0 and P-100 powders were obtained by adding large amount of CBZ into the S-0 and S-100 solution and then drying in vacuum oven for 24 h. Furthermore, the precursor (S-0 and S-100) was spin-coated onto a glass substrate. During spinning, CBZ (0.75 mL) was dripped at the center of the substrate and then the film was crystallized at 100°C for 20 min. The corresponding perovskite films were named as F-0, F-100 films, respectively.

The pure DMF (S-0) solution precursor lead to the rod shaped structure of MAPbI₃, which resulted from the different crystal growth rate between MAI and PbI₂ [21]. In contrast, the rod-like structure of MAPbI₃ disappeared if washed with non-polar solvent of CBZ during spinning the S-0 solution (Fig. 1a, b). Although washing with CBZ could improve the quality of MAPbI₃ films, pinholes still could be observed, which agreed well with previous reports [21,22]. Similarly, as shown in Fig. 1c, d, when no CBZ was applied during pure DMSO (S-100) solution precursor spinning process, large gaps existed in perovskite films. The resulted perovskite film with large gaps might be caused by

Figure 1 Planeview SEM topography for the perovskite films prepared by (a, b) S-0 solution and (c, d) S-100 solution. During the film spinning, the substrates (b) and (d) were treated with CBZ.

the large amount of DMSO in the precursor films. In comparison, the S-100 solution precursor spinning followed by CBZ drop-casting lead to smooth and homogeneous perovskite layers. These results indicate that washing with CBZ can control the evaporation rate of DMF or DMSO solvent. The excessive DMSO being washed off by CBZ produces more smooth and homogeneous MAPbI₃ films since DMSO has stronger coordinate ability with MAPbI₃ than DMF [23].

To further reveal the effect of CBZ on the perovskite crystallization process during the S-0 solution spinning, FTIR of DMF solvent, S-0 solution and P-0 powder were measured respectively. From Fig. 2a, b, we can see that the stretching vibrations of C=O are located at 1661 cm⁻¹ in DMF solvent and S-0 solution, but shifted to lower wavenumber of 1640 cm⁻¹ in the P-0 powder, which is originated from the weak C=O bond strength interaction with

PbI₂. Furthermore, N-H stretching and bending reveal that the P-0 powder includes MAI molecules [16,24]. From these results, it can be seen that no adduct formation in S-0 solution in absence of CBZ, while MAI-PbI₂-DMF adduct resulted from CBZ, which will retard the crystallization of MAI and PbI₂ during the annealing. These results are in good agreement with the results observed by SEM (Fig. 1a, b).

Unstable MAI-PbI₂-DMF intermediate has been recognized as the main reason for the pinhole on the perovskite film [21]. Therefore, evolution of the FTIR for DMSO solvent, S-100 solution and P-100 powder were further investigated. As shown in Fig. 3, the S=O bond strength is located at 1042 cm⁻¹ in DMSO solvent, but shifted to lower wavenumber of 1018 cm⁻¹ for the S-100 solution and P-100 powder due to the interaction with the Lewis acid Pb²⁺ [21,25]. Moreover, the FTIR spectra of MAI-DMSO, PbI₂-DMSO and S-100 solution are provided in Fig. S1 for the functional group comparison. Although N-H stretching and bending could not be found in FTIR spectra of MAI-DMSO and S-100 Solution, it appears in P-100 powder which indicates the formation of MAI-PbI₂-DMSO adduct [16,24]. In other words, PbI₂-DMSO adduct exists in S-100 solution and transforms into MAI-PbI₂-DMSO adduct after CBZ addition, which will inevitably influence the quality of perovskite films. The PbI₂-DMSO adduct in S-100 solution leads to large gaps on the perovskite film while the MAI-PbI₂-DMSO adduct results in smooth and homogeneous perovskite layer. FTIR spectra of other samples, such as, S-15, S-30, S-70 and S-85 are also provided in Fig. S2 to compare the functional group. The PbI₂-DMSO intermediate adduct appears even at a low volume ratio of DMSO (S-15), and becomes more apparently with the DMSO ratio increasing, further confirming that DMSO has stronger coordination ability with PbI₂ than DMF.

Figure 2 The FTIR of (a) DMF solvent, S-0 solution, P-0 powder and (b) expanded fingerprint region for the C=O vibrations.

Figure 3 The FTIR of (a) DMSO solvent, S-100 solution, P-100 powder, and (b) expanded fingerprint region for the S=O vibrations.

Figure 4 XRD patterns of (a) P-100 intermediate phases (b) and F-100 films. (c) Schematic models of the forming process of perovskite based on DMSO solvent.

On the basis of the above analysis, we can conclude that the MAI-PbI₂-DMF and MAI-PbI₂-DMSO intermediate phases play an important role in the crystal growth and result in pinhole and uniform thin films, respectively. Thus, P-0 and P-100 powders were analyzed through XRD measurement and compared with PbI₂ to understand the effect of underlying intermediate phase on the crystallization of perovskite films. The MAI, PbI₂, PbI₂-DMF and PbI₂-DMSO powers were characterized using XRD, and the reflection pattern is presented in Fig. S3. A strong peak appearing at 9.44° (PbI₂-DMSO) may be related to

the presence of DMSO molecules in PbI₂. Furthermore, the diffraction peaks at low angles (6.55° and 7.23°, Fig. 4a) appear at P-100 intermediate phases. According to previous reports, the lower angle of intermediate phases indicates that MAI and DMSO molecules have intercalated into the PbI₂ layered structure [18]. Based on the Bragg formulation, XRD peak of PbI₂ (Fig. S3) at 12.66° indicates that the interlayer spacing of the PbI₂ (001) layered structure is approximately 6.98 Å, while in the MAI-PbI₂-DMSO intermediate phases, the PbI₂ lattice translation vector along the *c* axis further increases. Af-

ter heating at 100°C for 20 min, the DMF and DMSO molecules inside the adducts tend to evaporate to produce perovskite. This mechanism could help us to understand the impact of intermediate phases on perovskite crystal growth. Namely, the crystallinity of intermediate phases is a crucial parameter on perovskite films [26,27]. The diffraction peak intensity of the MAI-PbI₂-DMSO is much higher than that of MAI-PbI₂-DMF intermediate phases. Consequently, XRD peak intensity of the MAPbI₃ film induced by MAI-PbI₂-DMSO is also much higher than that of MAPbI₃ film induced by MAI-PbI₂-DMF intermediate phases.

Analyses of these intermediate phases by SEM exhibit strikingly different morphologies. Fig. 5a shows fractured P-0 intermediate phase formed in the S-0 precursor solution, which easily loses DMF to transform MAPbI₃. In contrast, S-100 solution produces P-100 fibers that grow preferentially along fixed directions (Fig. 5b). Considering the process of shrinking during annealing, only the dense intermediate phase could lead to the formation of perovskite films without pinholes and cracks (Fig. 1d). Thus, the perovskite films produced by MAI-PbI₂-DMSO intermediate phase reveal full surface coverage.

Fig. 6a shows the typical *J*-*V* curves for perovskite solar cells based on S-0 and S-100 solutions with and without drop-casting CBZ, respectively. Through comparing the

photovoltaic parameters (J_{sc} , V_{oc} , FF and PCE) followed by addition of CBZ, a remarkable increase in J_{sc} and V_{oc} was observed, which can be attributed to the realization of dense films. In addition, Since the S-0 and S-100 solutions could tune the grain morphology and might have a great effect on the device performance, here, perovskite solar cells were prepared by various solutions and the photovoltaic properties were investigated (Table 1). The perovskite device fabricated with S-0 solution washed with CBZ exhibits a PCE of 9.44% with the V_{oc} of 0.93 V, the J_{sc} of 17.3 mA cm⁻², and FF of 58.7%. In contrast, the S-100 solution washed with CBZ derived perovskite solar cell shows a significantly enhanced PCE of 16.9% with V_{oc} = 1.06 V, J_{sc} = 20.6 mA cm⁻², and FF = 74.4%. We endow this to the pinhole-free film morphology of the F-100 film. The smooth film would prevent direct contact between the Spiro-OMeTAD and TiO₂. The corresponding incident photon-to-current efficiency (IPCE) for these cells further confirmed the J_{sc} results (Fig. 6b). In addition, Fig. S4 shows the typical *J*-*V* curves for the perovskite solar cells base on S-0 and S-100 solutions measured by forward scan (FS) and reverse scan (RS), respectively. The *J*-*V* curves of the forward and reverse scans (S-0 and S-100 solutions) were well coincident. These results reveal that the efficiency of the as-prepared perovskite solar cells is independent on the scanning direction. It is difficult to

Figure 5 SEM images of (a) P-0 and (b) P-100 intermediate phases.

Figure 6 *J*-*V* curves of typical cells based on S-0 and S-100 solutions and (b) the corresponding IPCE spectra for the typical cells.

Table 1 Photovoltaic properties of the perovskite solar cells prepared by various solutions

Solutions	J_{sc} (mA cm ⁻²)	V_{oc} (V)	FF (%)	PCE (%)
S-0	15.8	0.84	58.2	7.77
S-0+CBZ	17.3	0.93	58.7	9.44
S-100	16.5	0.99	72.4	11.8
S-100+CBZ	20.6	1.06	77.4	16.9

evaluate true device performance through the appearance of J - V curves. Therefore, steady state output of best solar cell was further measured. As shown in Fig. S5, the typical solar cell exhibits stable steady state output during 100 s at a bias of 0.86 V.

CONCLUSION

In summary, the effects of non-polar solvent (CBZ) on transformation of the intermediate phases were investigated. The high-quality perovskite films should be attributed to the rapid-transformed intermediates induced by non-polar solvent. The perovskite films grown using CBZ show dense surface coverage than the $\text{CH}_3\text{NH}_3\text{PbI}_3$ layers without washing with CBZ. Through studying the surface topography and crystal structure of different adducts, we revealed that strong polar solvent of DMSO constituted MAI-PbI₂-DMSO intermediate phase exhibited more neat structure, and the corresponding annealed films were uniform and dense. Consequently, the perovskite solar cell based on the MAI-PbI₂-DMSO intermediate leads to a high PCE value up to 16.9%. We hope this work will provide a new insight to understand the solvent engineering technology, and lead to perovskite solar cells with much higher PCE in the future.

Received 27 February 2017; accepted 29 March 2017;
published online 17 April 2017

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Acknowledgments This work was supported by the National Basic Research Program of China (2016YFA0202400 and 2015CB932200), the National Natural Science Foundation of China (21403247), the External Cooperation Program of BIC, Distinguished Youth Foundation of Anhui Province (1708085J09), Chinese Academy of Sciences (GJHZ1607), and STS project of Chinese Academy of Sciences (KFJ-SW-ST5-152).

Author contributions Ren Y performed the main experiments and wrote this manuscript. Zhu J directly guided and conducted this research including the design, modifying and polishing work related to this manuscript. Dai S supervised the projects and carefully reviewed and modified this manuscript. Duan B, Xu Y, Huang Y, Li Z, Hu L, Wang H and Hayat T 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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一步旋涂法中反溶剂对中间相演变的新视角

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摘要 反溶剂技术可以有效改善钙钛矿薄膜的质量. 本文揭示了氯苯 (CBZ) 在结晶过程中起的重要作用. 通过研究前体溶液中配合物的结构变化, 我们观察到, 在滴加反溶剂之前, MAI-PbI₂-DMF或MAI-PbI₂-DMSO尚未形成. 快速去除多余溶剂后形成中间相, 延缓钙钛矿结晶是得到高质量钙钛矿薄膜的关键. 其中MAI-PbI₂-DMSO中间体表现出了高结晶的棒状结构, 因此最终转变成非常致密和均匀的钙钛矿薄膜.