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Communication

## Preparation of $\text{CH}_3\text{NH}_3\text{PbI}_3$ thin films for solar cells via Vapor Transfer Method

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## ABSTRACT

Organometal halide perovskite based solar cells have emerged as one of the most promising candidates for low-cost and high-efficiency solar cell technologies. Here a Vapor Transfer Method (VTM) is used to fabricate high quality perovskite thin films in a balanced vacuum capsule. By adjusting the reaction temperature,  $\text{CH}_3\text{NH}_3\text{I}$  saturated vapor which then reacts with  $\text{PbI}_2$  films can be controlled and the formation process of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  perovskite films can be further influenced. Prepared perovskite films which exhibit pure phase, smooth surface and high crystallinity are assembled into planar heterojunction inverted solar cells. The whole fabrication process of solar cell devices is organic solution free. Finally, the champion cell achieved power conversion efficiency (PCE) of 13.08% with negligible current–voltage hysteresis under fully open-air conditions. The photovoltaic performance could be further enhanced by optimizing perovskite composition and the device structure.

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In the past few years, organometal halide perovskite (e.g.  $\text{CH}_3\text{NH}_3\text{PbX}_3$ , X = halogen) based solar cells have made great progress in the field of solar energy conversion. The power conversion efficiency (PCE) has skyrocketed to a certified 22.1% from 3.8% in 2009 on account of extraordinary optoelectronic properties and improved fabrication techniques [1,2]. Organometal halide perovskite is regarded as an outstanding absorber for the photovoltaic application, featuring with high extinction coefficient, excellent ambipolar charge mobility, small exciton binding energy and tunable band gap [3–5]. In order to fabricate perovskite films with desired properties and device performance, it is critical to achieve fine control over the fabrication process.

Since Snaith et al. fabricated the first simple planar heterojunction containing perovskite as absorber [6], planar p–i–n heterojunction has become the main-stream structure due to simple fabrication method and low processing cost [7–9]. Under this kind of planar-type architecture, incomplete and non-uniform coverage are usually observed [10], considerable research efforts have been devoted to exploring and improving fabrication techniques during the perovskite preparation process. Solution process and vapor deposition are two of the most widely used perovskite synthesis strategies. The former method like spin coating and spray coating is fairly simple and inexpensive [11–13], while the latter method is suitable for a wide variety of rigid and flexible applications

and can be used in large-scale production [14], including vacuum co-deposition [15,16], vapor-assisted solution process (VASP) [10,17], closed space vapor transport (CSVST), etc. [18]. Traditionally, the growth of perovskite is quite rapid in the solution process which usually results in thin films with pinhole formation and incomplete surface coverage [19]. On the other hand, it may take one or two hours when VASP method [16] is applied.

Here, we demonstrate the use of Vapor Transfer Method (VTM) to fabricate  $\text{CH}_3\text{NH}_3\text{PbI}_3$  ( $\text{MAPbI}_3$ ) perovskite thin films and subsequently solar cells with planar type structure. VTM is a moderate way to prepare perovskite films. The synthesis of  $\text{MAPbI}_3$  thin films takes about 15 min in our experiment. Organic and inorganic reaction precursors are sealed in a vacuum capsule (Fig. 1a), so that the reaction is isolated from the outside environment. Stable and balanced condition in the capsule is beneficial for the formation of stoichiometric perovskite films. The whole fabrication process is free from organic solution which may degrade the perovskite [20]. VTM is also compatible with other vacuum processes and could be used in large-scale solar energy production. Without using a glove box, samples were exposed to the air a few times and tested under a fully open-air condition, but the prepared perovskite films still demonstrated high quality and exhibited great optoelectronic properties.

As a sequential deposition process, we employed thermal evaporation method to fabricate phase-pure and uniform  $\text{PbI}_2$  films. The thickness of  $\text{PbI}_2$  thin film was controlled around 150 nm by a quartz thickness monitor. To fabricate uniformly distributed

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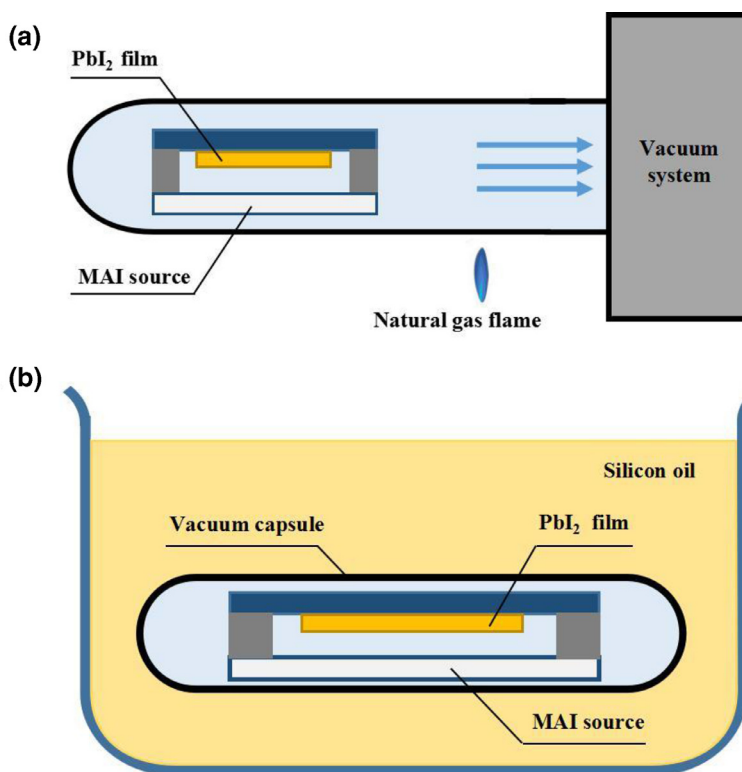


Fig. 1. Schematic representation of (a) the sealing process and (b) the synthesis process of perovskite films.

52  $\text{CH}_3\text{NH}_3\text{I}$  (MAI) source, mesoporous  $\text{TiO}_2$  layer was used to contain  
 53 MAI so as to spread the source uniformly in the mesoporous  
 54 structure. MAI powder was dissolved into DMF solution  
 55 ( $\sim 9 \text{ mg mL}^{-1}$ ) and dipped into mesoporous  $\text{TiO}_2$  layer which  
 56 was pre-fabricated on the slide glass by screen printing method  
 57 [21]. After being heated in air at  $60^\circ\text{C}$  on a hotplate for 30 min,  
 58 organic solution was evaporated and MAI powder remained in  
 59 the mesoporous structure. As-formed MAI source combined with  
 60 pre-sublimed  $\text{PbI}_2$  film sample was face to face put into a glass  
 61 tube. The distance between  $\text{PbI}_2$  thin film and MAI source in the  
 62 capsule was about 2 mm. As shown in Fig. 1(a), the glass tube  
 63 containing samples was connected with vacuum system. When the  
 64 pressure in the tube was decreased to  $5 \times 10^{-3} \text{ Pa}$ , a natural gas  
 65 flame was used to heat and seal the glass tube. As the pressure  
 66 in the tube was quite low, the air would extrude melted glass  
 67 and seal the tube as a capsule. After that, the capsule was heated  
 68 at  $120^\circ\text{C}$  in an oil bath to guarantee temperature uniformity as  
 69 illustrated in Fig. 1(b). MAI saturated pressure in the vacuum  
 70 capsule increased with the temperature. As a result, MAI vapor  
 71 filled the entire capsule and reacted with  $\text{PbI}_2$  film gradually.  
 72 In the present experiment condition, 15 min is enough to form  
 73 perovskite films completely.

74 As perovskite films were fabricated successfully using our  
 75 method, a simple solar cell structure without buff layers was se-  
 76 lected to test the photovoltaic performance. The structure of en-  
 77 tire device and energy diagram of each layer are depicted in Fig.  
 78 2(a) and (b), respectively [22,23]. On top of ITO-coated glass,  
 79 perovskite layer ( $\text{MAPbI}_3$ ) was sandwiched between PEDOT:PSS and  
 80 fullerene layer. Ag was used as the top electrode. To avoid solvent  
 81 residual and chemical impurity,  $\text{C}_{60}$  was selected as the electron-  
 82 transporting layer (ETL) and deposited by thermal evaporation,  
 83 although PCBM and ICBA may be more effective [24]. Consequently,  
 84 the whole fabrication process of the solar cell devices was free  
 85 from organic solution.

86 The glass/ITO substrates were cleaned with acetone, alcohol and  
 87 deionized water successively in an ultrasonic cleaner for 30 min  
 88 and were blow-dried using nitrogen gas. PEDOT:PSS was spin-  
 89 coated on the patterned ITO under 3000 rpm for 60 s and annealed  
 90 at  $130^\circ\text{C}$  for 20 min in air.  $\text{MAPbI}_3$  perovskite absorbers were de-  
 91 posited on the PEDOT:PSS/ITO/glass substrates by VTM mentioned  
 92 above.  $\text{C}_{60}$  ( $\sim 40 \text{ nm}$ ) and silver electrode were thermal evapo-  
 93 rated on the top of  $\text{MAPbI}_3$  films sequentially. The active area was  
 94  $5 \text{ mm}^2$  which was determined by the overlap of the anode and  
 95 cathode.

96 The scanning electron microscopy (SEM) images were obtained  
 97 by PHILIPS XL30FEG. The measurement was conducted under  
 98 20 kV. The atomic force microscopy (AFM) images were taken by  
 99 BRUKER Dimension edge. The images and surface roughness data  
 100 were given by NanoScopeAnalysis software. The X-ray diffraction  
 101 (XRD) was performed by BRUKER D8 ADVANCE. The photovoltaic  
 102 performance was measured by Kethley 2400 source meter under  
 103 AM 1.5 G sunlight irradiation.

104 As shown in Fig. 3(c), the color of samples gradually changes  
 105 from light yellow to dark brown during the formation process of  
 106 perovskite films. The XRD measurement was also performed in  
 107 ambient condition (60% humidity level) to investigate the forma-  
 108 tion of perovskite. In Fig. 3(a), the XRD pattern clearly shows the  
 109 transformation process from  $\text{PbI}_2$  to  $\text{MAPbI}_3$ . The sample is com-  
 110 posed of  $\text{PbI}_2$  at the initial stage. With time evolution,  $\text{PbI}_2$  reacts  
 111 with MAI vapor and turns into  $\text{MAPbI}_3$ . As the perovskite phase  
 112 gradually appears, the intensity of  $\text{PbI}_2$  phase reduces until com-  
 113 pletely disappears after reacting at  $120^\circ\text{C}$  for 15 min. Further pro-  
 114 longing the reaction time does not seem to make effect on the  
 115 as-formed  $\text{MAPbI}_3$  films. The detailed XRD characterization of the  
 116 sample reacted at  $120^\circ\text{C}$  for 15 min is shown in Fig. 3(b). A set of  
 117 strong sharp diffraction peaks at  $14.28^\circ$ ,  $28.53^\circ$ ,  $42.96^\circ$  and  $58.73^\circ$   
 118 corresponding to the (110), (220), (330) and (440) crystal planes  
 119 of  $\text{MAPbI}_3$  appears [4], indicating great crystallization and high

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