Research Article

Improving the Morphology of the Perovskite Absorber Layer in Hybrid Organic/Inorganic Halide Perovskite MAPbI₃ Solar Cells

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Recently, perovskite solar cells have attracted tremendous attention due to their excellent power conversion efficiency, low cost, simple fabrications, and high photovoltaic performance. Furthermore, the perovskite solar cells are lightweight and possess thin film and semitransparency. However, the nonuniformity in perovskite layer constitutes a major setback to the operation mechanism, performance, reproducibility, and degradation of perovskite solar cells. Therefore, one of the main challenges in planar perovskite devices is the fabrication of high quality films with controlled morphology and least amount of pin-holes for high performance thin film perovskite devices. The poor reproducibility in perovskite solar cells hinders the accurate fabrication of practical devices for use in real world applications, and this is primarily as a result of the inability to control the morphology of perovskites, leading to large variability in the characteristics of perovskite solar cells. Hence, the focus of research in perovskites has been mostly geared towards improving the morphology and crystallization of perovskite absorber by selecting the optimal annealing condition considering the effect of humidity. Here we report a controlled ambient condition that is necessary to grow uniform perovskite crystals. A best PCE of 7.5% was achieved along with a short-circuit current density of 15.2 mA/cm², an open-circuit voltage of 0.81 V, and a fill factor of 0.612 from the perovskite solar cell prepared under 60% relative humidity.

1. Introduction

Organic-inorganic metal halide perovskites have recently shown great potential for application in solar cells with excitingly high performances with an up-to-date NREL-certified record efficiency of 22.1%, as reported by KRICT/UNIST in November 2016. Perovskite materials are currently being investigated because of their low cost materials, low cost fabrication techniques, and ability to act as efficient light-absorber layer for organic and inorganic photovoltaic applications. Perovskite absorber materials have the good optical, electrical, and semiconductor properties such as high diffusion length and high charge mobility. Another important factor that makes perovskite material highly sought after is the processing and manufacturing techniques; there are many of deposition techniques that can be used in the preparation of high quality thin films; these include one-step deposition, two-step sequential deposition, thermal evaporation, and vapor-assisted vacuum deposition techniques.

We have seen different studies focused on ways to control the morphology of perovskite films through proper selection of annealing conditions (temperature and humidity), perovskite precursor solution ratio, additives to side-chain of precursor solution, and solvent engineering. Huang et al. [1] earlier reported the optimal crystallization conditions for planar and mesoporous perovskite layer architectures as ~100°C with annealing duration ranging from 30 to 120 minutes. Salim et al. [2] discussed the impact of morphology and device architecture on device performance in perovskite-based solar cells. Eperon et al. [3] presented a morphological control scheme for high performance, solution-processed planar heterojunction perovskite solar cells, while Gong et al. [4] proposed a controllable perovskite crystallization approach by water additive for high performance solar cells. Hsu et al. [5] proposed a two-step thermal annealing process to improve the morphology of spin-coated films for highly efficient perovskite hybrid photovoltaics, while Dualeh et al. [6] and Saliba et al. [7] investigated the effects of

thermal processing protocol and annealing temperature on film morphology and crystallization of organic–inorganic hybrid perovskite solar cells. Tidhar et al. [8] discussed the crystallization of methyl ammonium lead halide perovskites and its implications in photovoltaic applications. Grancini et al. [9] also studied the impacts of the crystallization processes on the structural and optical properties of hybrid perovskite films. Liang et al. [10] presented additive enhanced crystallization of solution-processed perovskite for highly efficient planar heterojunction solar cells.

In order to determine the optimal crystallization conditions, different experiments and characterization techniques (such as Scanning Electron Microscopy, current density-voltage measurements, and incident photon-to-current efficiency measurements) have been adopted so as to properly interpret the results from experiments and fabrication. After interpreting the results from characterization data, we observed that poor perovskite crystallites translate to low power conversion efficiencies and more pin-hole defects are observed in the perovskite film.

2. Experimental Section

2.1. Materials. Indium tin oxide coated with polyethylene terephthalate film (ITO/PET) with 12 ohm/sq surface resistivity, patterned indium tin oxide coated glass slide (with 8 ohm/sq surface resistivity), VWR microscope slides, poly(3,4-ethylenedioxythiophene)-polystyrene sulfonate (PEDOT:PSS) (1.3 wt.% dispersion in water), PEDOT content 0.5 wt.% and PSS content 0.8 wt.%, titanium oxide (TiO_2) 16 wt.%, 1,2-Dichlorobenzene (DCB), N,N-dimethylformamide (DMF), and titania paste (transparent) 19.0 wt.%, drierite (with indicator, 6 mesh), 0.50 μm Whatman filter paper, isopropyl alcohol, acetone, methylamine solution (33 wt.% in absolute ethanol, CH_3NH_2), hydroiodic acid (57 wt.% distilled, HI), lead(II) iodide (99.99%), and conductive silver printing ink (Ag paste) with resistivity range 5–6 $\mu\Omega\text{cm}$ were ordered from Sigma-Aldrich and used as received without further modification or purification.

2.2. Perovskite Precursor Synthesis. Preparing the precursor perovskite solution is the first step in order to produce a perovskite solution and perovskite film. Methylammonium iodide ($\text{CH}_3\text{NH}_3\text{I}$) was prepared by reacting methylamine (CH_3NH_2) solution in absolute ethanol with hydroiodic acid (HI) (based on the steps and quantities stated in literature) [11]. To produce MAPbI_3 precursor solution, methylammonium iodide (MAI) and PbI_2 were dissolved in anhydrous N,N-dimethylformamide (DMF) in a 1:1 molar ratio, at 0.88 M of each in a nitrogen-filled box to yield 0.88 M perovskite precursor solution.

2.3. Perovskite Thin Film Preparation. The MAPbI_3 films were prepared by spin-coating the derived precursor solution in a nitrogen-filled glovebox at 2500 rpm for 40 s on top of PEDOT:PSS/ITO/PET substrate. The resulting film was then annealed at 85°C for about 2 hours in a controlled humidity chamber. The thickness of MAPbI_3 perovskite film

was controlled by varying the spin-speed and also using varying precursor concentrations.

2.4. Device Fabrication. The perovskite solar cell devices (PSCDs) were fabricated on ITO glass substrates; the ITO substrate was partly etched with 1M HCl acid so as to correctly allow for electrode contact placement and prevent short-circuit. The ITO substrates were initially precleaned with cleansing soap and sonicated in deionized water, acetone, and isopropanol for 15 minutes. The substrates were dried in vacuum oven and thereafter allowed to undergo oxygen plasma treatment for 15 minutes. PEDOT:PSS solution was filtered using Whatman filter paper in order to remove aqueous colloidal particles in the original solution. The filtered PEDOT:PSS solution was spun on the ITO substrate at 2500 rpm for 45 seconds and annealed at 120°C for 30 minutes. After that, the perovskite layer is prepared by spin-coating the perovskite solution on the top of PEDOT:PSS in a nitrogen-filled glove box and transferred to a controlled humidity chamber where it is annealed at 85°C for 2 hours. TiO_2 (an electron transporting layer material) is then spin-coated on the perovskite film layer at 2500 rpm for 30 s in a N_2 -filled glovebox. After spin-coating, the film was allowed to dry for about 18 hours. Ultimately, approximately 110 nm thick silver electrode was thermally deposited on the top of TiO_2 layer in a vacuum with a base pressure of 0.000008 mbar and this step completes the fabrication of the PSCD. Throughout the entire device fabrication, we kept other process parameters constant while we varied humidity conditions in the chamber necessary for annealing the perovskite absorber layer.

2.5. Characterizations. The current density-voltage (J - V) characteristics of PSCDs were measured using a Keithley 2400 source measurement unit scanning the devices at 100 mV/s and a Newport solar simulator with Air Mass 1.5 Global (AM1.5G) illumination and light intensity 100 mW/cm^2 . Investigating the surface morphology and cross sections of our selected devices were carried out using a field emission scanning electron microscope (FESEM, JEOL-7401). The incident photon-to-current conversion efficiency (IPCE) spectra was recorded over the wavelength from 300 to 800 nm. The devices fabricated were tested under ambient environmental conditions with controlled humidity level and also under vacuum condition. The active device area was about 0.1225 cm^2 after masking for J - V measurements.

2.6. Device Architecture. Generally, there are two device architectures that are often used in the fabrication of halide perovskite structures, namely, planar heterojunction architecture (PHJ) and mesoscopic architecture [12, 13]. In our device architecture, we considered the PHJ architecture for the PSCD structure glass/ITO/PEDOT:PSS/ MAPbI_3 / TiO_2 /Ag (structure is stacked from bottom to top); the structure is seen in Figure 1. The PHJ-PSCD structure provides an interface that makes it easy to investigate material properties in the absence of the mesoporous scaffold layer. The energy level (in eV) diagram of the corresponding

TABLE 1: Device performance parameters summary.

Conditions	Voc (V)	Jsc (mA/cm ²)	FF (%)	PCE (%)
Vacuum	0.75	13.3	47.1	4.7
30% RH	0.77	13.9	53.1	5.6
60% RH	0.81	15.2	61.2	7.5
90% RH	0.73	13.8	46.6	4.5

FIGURE 1: Device structure of the perovskite solar cell device.

FIGURE 2: Corresponding energy level diagram in eV of the materials used in the perovskite solar cells.

materials used in the perovskite solar cells is depicted in Figure 2. The light source is shone from the glass/ITO end and it is important that the PEDOT:PSS layer is able to transmit the incoming light efficiently because this directly affects the generated current density. The transmittance of PEDOT:PSS is determined by the concentration of the spin-coated precursor solution; hence, it is important that we do not use a too high or too low PEDOT:PSS precursor solution.

3. Results and Discussion

The PHJ perovskite device structure is based on the p-i-n structure of ITO/PEDOT:PSS/MAPbI₃/TiO₂/Ag. The ITO layer acts as the anode, PEDOT:PSS as the transparent and conductive layer for hole transport, MAPbI₃ as the absorber material, TiO₂ as the electron transport layer, and Ag as the cathode layer. The *J-V* characteristics of the fabricated perovskite devices are shown in Figure 3(a). Table 1 shows the device performance of the four perovskite devices fabricated in four different humidity conditions. The perovskite device with the absorber material annealed at 60% relative humidity (RH) showed high photovoltaic (PV) parameters with the maximum power conversion efficiency (PCE) of 10 device samples reaching 7.5%. The device annealed under vacuum, 30% humidity, and 90% humidity conditions demonstrated efficiencies of 4.7%, 5.6%, and 4.5%, respectively. Further experiments were conducted to specifically test 10 perovskite devices annealed under 60–70% relative humidity before having to conclude that the optimal relative humidity was at 60%. The maximum Voc (open-circuit voltage) and Jsc (short-circuit current density) are 0.81 V and 15.2 mA/cm². The largest fill factor derived was 61.2%. According to Gangishetty et al. [14] low relative humidity will cause large gaps between perovskite crystallites and consequently leads to hysteresis that affects the overall performance of the device.

From Table 1, we observe a huge variation in the fill factor (FF) which can be attributed to the series resistance

TABLE 2: Device performance parameters summary for 20 devices.

Conditions	Voc (V)	Jsc (mA/cm ²)	FF (%)	PCE (%)
Vacuum	(1) 0.75	13.30	47.1	4.70
	(2) 0.77	13.10	46.5	4.69
	(3) 0.68	12.95	48.1	4.24
	(4) 0.70	13.02	47.5	4.33
	(5) 0.75	13.25	47.0	4.67
30% RH	(6) 0.75	14.00	53.2	5.58
	(7) 0.75	13.95	53.2	5.57
	(8) 0.78	14.20	50.1	5.54
	(9) 0.78	14.30	50.1	5.58
	(10) 0.77	13.90	53.1	5.60
60% RH	(11) 0.81	15.20	61.2	7.50
	(12) 0.78	14.55	61.3	6.96
	(13) 0.80	15.00	62.2	7.46
	(14) 0.80	15.00	61.5	7.38
	(15) 0.80	14.67	61.0	7.16
90% RH	(16) 0.73	13.80	46.6	4.59
	(17) 0.70	13.70	46.8	4.48
	(18) 0.71	13.67	46.4	4.50
	(19) 0.65	13.40	49.5	4.31
	(20) 0.68	13.80	47.5	4.46

FIGURE 3

of the devices which is primarily due to the charge transport properties of the charge carrier properties of HTM [15]. The smaller resistance present in the 60% RH condition likely contributed to the higher FF value. On top of that, Voc values experience a big gap when compared with other conditions; this is very possible when fewer bimolecular recombinations occur as a result of reduced number of traps and grain boundaries [12]. The IPCE spectra supports

the evident increase in the short-circuit current density as seen in Figure 3(b). The measurements taken for IPCE were conducted over a spectral range of 300 nm to 800 nm.

The statistical distributions of the PCE of 20 fabricated perovskite device samples are shown in Figure 4. Table 2 also shows the different values of Voc, Jsc, FF, and PCE for 20 perovskite solar cell devices fabricated in vacuum, 30% RH, 60% RH, and 90% RH. Table 3 is the best cell performance

TABLE 3: Best device performance for different scan direction and scan rate.

Conditions	Scan direction	Scan rate (V/s)	Voc (V)	Jsc (mA/cm ²)	FF (%)	PCE (%)
Vacuum	Reverse scan	0.05	0.72	12.32	44.2	3.92
		0.1	0.74	12.57	45.0	4.19
		0.2	0.74	12.94	45.6	4.37
		0.5	0.75	13.30	47.1	4.70
	Forward scan	0.05	0.72	12.30	44.2	3.91
		0.1	0.74	12.55	44.9	4.17
		0.2	0.74	12.94	45.6	4.37
		0.5	0.75	13.29	46.9	4.67
30% RH	Reverse scan	0.05	0.73	12.65	51.6	4.76
		0.1	0.75	12.95	52.2	5.07
		0.2	0.76	13.45	52.8	5.39
		0.5	0.77	13.90	53.1	5.68
	Forward scan	0.05	0.73	12.62	51.1	4.71
		0.1	0.74	13.40	52.0	5.16
		0.2	0.75	13.45	52.5	5.30
		0.5	0.76	13.90	52.8	5.57
60% RH	Reverse scan	0.05	0.77	14.00	59.5	6.41
		0.1	0.79	14.28	60.1	6.78
		0.2	0.79	14.78	60.7	7.08
		0.5	0.81	15.20	61.2	7.50
	Forward scan	0.05	0.77	13.90	59.1	6.33
		0.1	0.77	14.25	60.0	6.58
		0.2	0.78	14.70	60.4	6.93
		0.5	0.81	15.19	60.8	7.48
90% RH	Reverse scan	0.05	0.70	13.10	45.2	4.14
		0.1	0.71	13.20	45.6	4.27
		0.2	0.72	13.45	46.0	4.45
		0.5	0.73	13.80	46.6	4.59
	Forward scan	0.05	0.69	12.80	44.5	3.93
		0.1	0.70	12.80	44.9	4.02
		0.2	0.71	13.35	45.4	4.30
		0.5	0.73	13.80	46.0	4.53

FIGURE 4: PCE distribution of twenty perovskite devices fabricated under different atmospheric conditions (vacuum, 30% RH, 60% RH, and 90% RH).

characteristics table for different humidity conditions with respect to scan direction and rate.

Figure 5 shows the PV characteristics properties of the MAPbI₃ hybrid perovskite solar cell. The *J*-*V* curve of the best performing MAPbI₃ hybrid perovskite solar cell is the one fabricated under 60% RH condition, Voc of 0.81 V, Jsc of 15.20 mA/cm², FF of 61.2%, and PCE of 7.5% in the reverse scan direction, and Voc of 0.81 V, Jsc of 15.19 mA/cm², FF of 60.8%, and PCE of 7.48% in the forward scan direction under standard illumination of 100 mW/cm² (AM1.5 global). According to the *J*-*V* curves, the MAPbI₃ hybrid perovskite cell showed a very small *J*-*V* hysteresis with respect to the scan direction and a significant *J*-*V* hysteresis is observed when the scan rate is varied from 0.05 V/s to 0.5 V/s.

The X-ray diffraction (XRD) patterns of the perovskite films under different annealing conditions (vacuum, 30% RH, 60% RH, and 90% RH) are shown in Figure 6. XRD spectra results for each film as deposited on ITO/PEDOT:PSS are reported in order to check the crystallinity of perovskites. The main diffraction peaks of the perovskite films are observed at 14.1° and 28.4°. A new diffraction peak is observed at

FIGURE 5: Continued.

FIGURE 5: J - V curves of the MAPbI₃ hybrid perovskite solar cell with respect to different scan directions (reverse and forward) and different scan rates (0.05 V/s, 0.1 V/s, 0.2 V/s, and 0.5 V/s).

FIGURE 6: XRD patterns of perovskite films annealed in different conditions (vacuum, 30% RH, 60% RH, and 90% RH).

12.6° in the XRD spectra of 90% RH; this can be attributed to the diffraction peak of PbI₂ formed during the decomposition of perovskite crystal structure when exposed to excess moisture and oxygen. The main difference observed in the other annealing conditions is simply the minimal increase in the peak intensity (from vacuum to 60% RH). This demonstrates that there is a boost in the crystallinity of MAPbI₃ films formed in the presence of increased relative humidity conditions.

The perovskite film was spin-coated using two-deposition technique [16]. Obtaining an excellent performance in PHJ perovskite devices requires that we obtain a quality film morphology by way of how the spin-coated film is annealed and crystallized. The rate of crystallization of perovskite

absorber film is one of the many difficult parameters to be directly controlled in the fabrication process; we decide to understand the crystallization behavior of the film by taking SEM images whereby we are able to understand the surface morphological features peculiar to the film under study. The crystallinity of the film influences factors such as diffusion length, hole and electron mobility, and charge dissociation efficiency [3, 17]. The crystallization behavior of perovskite material as seen in the results from SEM images shows that humidity is a major factor contributing to the way perovskite crystals are formed. Zhou et al. [18] showed that mixed halide perovskite when annealed under 30% RH was subject to a different conversion process; MAPbI₃ phase could be observed distinctly in the presence of moisture.

FIGURE 7: SEM images of perovskite absorber in (a) vacuum, (b) 30% RH, (c) 60% RH, and (d) 90% RH.

Figure 7 shows the SEM images of perovskite (MAPbI_3) formed by two-step deposition of PbI_2 and the precursor solution. Here we show the annealing treatment for the perovskite absorber carried out at different relative humidity (0%, 30%, 60%, and 90%). We observed the formation of cuboid crystals across all the humidity conditions but varying number of pin-holes (trap states). The greatest number of pin-holes is seen in the 90% RH, next to that is vacuum, next to that is 30% RH, and finally with the least number of pin-holes is 60% RH. There is a linear relationship existing between the number of pin-holes in the film and also the power conversion efficiency of the perovskite device; this explains the reason for high efficiency in the 60% RH condition (because the cuboid crystals are tightly packed with very few gaps present).

4. Conclusion

In this work, we developed an effective condition for annealing perovskite absorber after spin-coating using humidity control experimental methods to determine the effect of environmental conditions (humidity and moisture) on the perovskite film and the performance of the organic-inorganic halide perovskite MAPbI_3 solar device. PEDOT:PSS is used as the HTM and TiO_2 as the ETM. It is important that we carefully select the transporting layers as this plays a vital role in optimizing the overall power conversion efficiency and performance. It was found out that the efficiencies of the PSCDs are highly dependent on the crystallization and annealing environment of the perovskite absorber, as the relative humidity falls in the range ~60–65%; we observe a significant improvement in the morphology of MAPbI_3 due

to formation of more uniform perovskite crystallites with fewer grain boundaries.

Additionally, efficiency improvements can be achieved through proper selection of absorber, HTL, and ETL materials and cathode/anode electrode material, controlling film thickness, and interface engineering. Efficiency of up to 7.5% was achieved using a normal PHJ structure solar cell. The results presented show a promising progress towards achieving high-efficiency photovoltaic device applications.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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