Activation of Weak Monochromic Photocurrents by White Light Irradiation for Accurate IPCE Measurements of Carbon-Based Multi-Porous-Layered-Electrode Perovskite Solar Cells

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ABSTRACT
Variations in the time courses of the activation of fully-printable carbon-based multi-porous-layered-electrode perovskite solar cells (MPLE-PSCs) can lead to differences between the photocurrent density \(J_{sc}\) values obtained from one-sun photocurrent density-voltage \((J–V)\) measurements and from incident-photon-to-current efficiency \((IPCE)\) integration when using monochromic light. In the present work, the \(J_{sc}\) calculated from IPCE data was initially equal to half that obtained from one-sun \(J–V\) measurements. However, equivalent values were obtained when the \(J–V\) measurements were performed after 10 min of irradiation by a white light LED from the side of the device. This finding will be very important with regard to permitting accurate photovoltaic evaluation of MPLE-PSCs in the future.

Keywords: Perovskite Solar Cells, Carbon-Graphite Electrode, Mesoporous Metal Oxide, \(J–V\) and IPCE Measurement

1. Introduction
The photoactivation of standard thin-film planar perovskite solar cells (TF-PSCs) by exposure to light has been previously researched.\(^1\)\(^-\)\(^10\) However, in the present study, we found that the photoactivation timing of the photocurrent density \(J_{sc}\) using carbon-based multi-porous-layered-electrode PSCs (MPLE-PSCs) differs depending on whether current density–voltage \((J–V)\) or incident-photon-to-current efficiency \((IPCE)\) data are acquired, which is different from standard TF-PSCs.

The MPLE-PSCs, first reported by Han’s group in 2013,\(^10\) have been widely investigated because they are highly stable, permit facile fully-printable fabrication and are made of cost-effective Earth-abundant materials. These devices do not require an expensive hole transport layer (HTL) materials such as Spiro-OMeTAD (Figs. 1a and b).\(^11\) HTL-free carbon-based MPLE-PSCs comprise an electrode with a scaffold layer structure consisting of an electron transport layer (ETL) material such as mesoporous titanium oxide \((m-TiO_2)\), a spacer layer (such as mesoporous zirconium oxide; \(m-ZrO_2)\) and a counter electrode layer made of graphite. In the fabrication of these devices, a perovskite precursor solution is drop-cast on top of the carbon layer such that the entire scaffold layer is filled with perovskite to complete the device. To obtain optimal performance from this architecture, the mesopores of the scaffold layer must be completely filled with perovskite, and one effective means of doing so is to apply a mixture of lead iodide \((PbI_2)\) with methylammonium (MA) and 5-aminovaleric acid \((5-AVA)\) cations together with \(\gamma\)-butyrolactone \((GBL,\) acting as the solvent).\(^11\) The resulting mixed dimension \((2D/3D)\) perovskite crystals having the formula \((5-AVA)_x(MA)_{1-x}PbI_3\) have shown high performance due to low defect concentrations and improved pore filling.\(^11\) In addition, this architecture has demonstrated exceptional performance stability because the thick graphite layer protects the internal perovskite from ambient air and humidity.\(^15\)\(^-\)\(^16\) MPLE-PSCs have also received

![Figure 1](image_url)
a great deal of attention because these devices have exhibited photoelectric conversion efficiency values up to 17%,\textsuperscript{17} can be manufactured at low cost, and are extremely stable.

However, the \( J_{sc} \) values obtained from integrating IPCE data are typically lower than those obtained from one-sun irradiation experiments (or are not reported).\textsuperscript{18–23} This is unfortunate, because good agreement between the \( J_{sc} \) values obtained from \( J-V \) measurements and IPCE calculations is quite important with regard to confirming the accuracy of these values. Theoretically, the \( J_{sc} \) values obtained from \( J-V \) curves should be consistent with those calculated from IPCE data, and the correct evaluation of photo-voltaic performance can be confirmed by comparing the \( J_{sc} \) values from these two methods.\textsuperscript{24} The aim of the present work was therefore to introduce an accurate means of finding \( J_{sc} \) values for MPLE-PSCs.

2. Experimental

2.1 Materials

The device employed in this work was fabricated according to procedures previously reported in the literature.\textsuperscript{11,25,26} All the materials employed in this study were used as received without any purification. These included PbI\(_2\) (99.99), MA iodide (MAI, 98.0\%), Tokyo Chemical Industry Co., Ltd.), 5-ammonium valeric acid iodide (5-AVAI, Greatcell Solar), GBL (electrochemistry grade, Kanto Chemical Co., Inc.), titanium diisopropoxide bis(acylacetone) (75 wt.% in isopropanol, Sigma-Aldrich), titanium (IV) dioxide paste (SnO\(_2\) PST-30NDR, JGC Catalysts and Chemicals, Ltd.), zirconium dioxide paste (ZrO\(_2\), Zr-Nanokide ZT/SP, Solaronix SA), ethanol (99.5%, Hayashi Pure Chemical Ind., Ltd.), \( \alpha \)-terpineol (Kanto Chemical Co., Inc.) and fluoroine-doped tin oxide (FTO) glass substrates (TEC-15, Nippon Sheet Glass-Pilkington Co., Ltd.). The carbon paste was made of pyrolytic graphite and was prepared using a modification of a previously reported procedure.\textsuperscript{27–30}

2.2 Preparation of the perovskite precursor solution

The precursor solution was prepared in a glove box filled with \( \text{N}_2 \) gas having a dew point between –20 and –18 °C. A 1.2 M solution of the (5-AVA\(_2\))(MA\(_2\))\(_x\)Pb\(_y\) perovskite precursor was made by mixing 0.5532 g PbI\(_2\), 0.1812 g MAI and 0.0147 g 5-AVAI in 1 mL of GBL followed by stirring at 60 °C and 800 rpm for 2 h.

2.3 Device fabrication

All device fabrication processes were performed under ambient air at room temperature (15 to 25 °C) and at relative humidity values from 30 to 50%. The conductive layer of the FTO glass (100 × 40 mm\(^2\)) was separated by etching and ultrasonically cleaned with a detergent solution (1%, white 7-AL, Yuai Kasei) and with ethanol for 15 min each. A blocking layer made of compact TiO\(_2\) (c-TiO\(_2\)) was subsequently deposited on the patterned substrates by spray pyrolysis at 500 °C, employing a hot plate and applying 0.66 mL titanium diisopropoxide bis(acylacetone) solution in 22.5 mL ethanol. In this process, the spray was applied evenly over the entire hot plate for a period of 10 s. A 0.55 mm mesoporous TiO\(_2\) (m-TiO\(_2\)) layer was then screen printed using a paste prepared by mixing the TiO\(_2\) paste with \( \alpha \)-terpineol in a 1:2 mass ratio, after which the specimen was held at room temperature for 5 min and then dried on a hot plate at 125 °C for 15 min. A mesoporous ZrO\(_2\) (m-ZrO\(_2\)) spacer layer (1.7-µm thick) was screen printed using the ZrO\(_2\) paste, after which the device was held at room temperature for 10 min and then dried on a hot plate at 125 °C for 15 min. The TiO\(_2\) and ZrO\(_2\) layers were annealed at 500 °C for 1 h in an electric furnace (applying a temperature rise time of 30 min and hold time of 30 min). Subsequently, a graphite layer (23-µm thick) was screen printed using a graphite paste made in our own laboratory, after which the specimen was held at room temperature for 5 min and then dried on a hot plate at 125 °C for 15 min. The graphite layer was annealed at 400 °C for 31 min (with a temperature rise time of 5 min and a hold time of 26 min) in an electric furnace. The substrate was then cooled to room temperature and cut into single cells, following which ultrasonic soldering was performed attach contacts to both electrode ends. The area around the scaffold layer was masked with heat resistant polyimide tape so that the perovskite solution would primarily fill the scaffold layers. Finally, 2.0 µL of the 1.2 M (5-AVA\(_2\))(MA\(_2\))\(_x\)Pb\(_y\) perovskite precursor solution was supplied to the scaffold layers by drop-casting. Each filled device was covered with a glass lid in a petri dish at room temperature for 30 min and then heated at 50 °C on a hot plate with a glass lid cover for 30 min. The cover was then removed and the device was dried at 50 °C for 1 h to complete the fabrication process.

2.4 Characterization

All assessments were carried out under ambient conditions. Cross-sectional scanning electron microscopy (SEM) images were obtained using a JSM-6510 instrument (JEOL). Energy dispersive X-ray spectroscopy (EDX) line scan and mapping analyses of the devices were also performed (TM3030, HITACHI). \( J-V \) curves were acquired with a DC voltage current source (BZ901A, Agilent) under a solar simulator (AM1.5G, 100 mW cm\(^{-2}\)) equipped with a 500 W xenon (Xe) lamp (YSS-100A, Yamashita Denso). The power of the AM1.5G solar simulator was calibrated using a reference Si photodiode (Bunkou-Keiki Co., Ltd., Japan). The area irradiated with light was 3 × 3 mm, with an active area on the device of 0.36 cm\(^2\) and a mask opening area of 0.09 cm\(^2\). The measurement voltage ranged from –0.1 to 1.0 V and both forward and reverse scans were applied with steps of 0.01 V, using a measurement delay of 40 ms. Each measurement was performed five times with an interval of 3 min between each measurement, and the device was continually illuminated between measurements. The AM1.5G light aging trials were performed under open-circuit conditions. IPCE spectra were acquired using a 150 W Xe lamp with a monochromator as a monochromatic light source (PEC-S20 IPCE Measurement Apparatus, Peccell Technologies, Inc.). Calibration with a Si photodiode was carried out prior to the IPCE measurements, and each measurement was performed five times at 3 min intervals. During each interval, the specimen was irradiated using an LED (ELPA, SOT-01C01, DC 3.5 V, 300 mA) from the side of the device to provide light-soaking. During these trials, the current was maintained at 0.2 µA, equivalent to 0.238 to 0.357% simulated solar intensity (AM1.5G). The light intensity values were derived from the equations

\[
I(\text{LED}) = 20 \text{ to } 30 \text{ [µA]} / 0.42 \text{ [cm}^2\text{]} = 0.0476 \text{ to } 0.0714 \text{ [mA cm}^{-2}\text{]},
\]

\[
I(\text{AM1.5G}) = 20 \text{ [mA cm}^{-2}\text{]},
\]

and

\[
I(\text{LED})/I(\text{AM1.5G}) = 0.0476 \text{ to } 0.0714 \text{ [mA cm}^{-2}\text{]} / 0.0476 \text{ [mA cm}^{-2}\text{]} \times 100 = 0.238 \text{ to } 0.357%.
\]

The LED light was switched off during the IPCE measurements, and the distance between the device and the LED light was fixed at 10 mm. Since the FF and PCE returned to their original values after leaving the cell in the dark following light activation, it is preferable to repeat this light-soaking until each parameter is again stabilized when assessing MPLE-PSCs.

3. Results and Discussion

Figure 2 shows cross-sectional SEM images and EDX data acquired from an <FTO/m-TiO\(_2\)/m-ZrO\(_2\)/carbon-graphite> elec-
trode before and after filling with the perovskite. The SEM image clearly shows the boundaries between each porous layer both with and without the perovskite (Figs. 2b and e). It is difficult to confirm the filling with the perovskite from the SEM image. However, the data from the EDX line scan indicate that Pb and I (due to the perovskite) were present in the perovskite-filled device, but not in the unfilled device (Figs. 2c and f). Similarly, the EDX mapping also confirms the filling of the mesoporous layer with perovskite (Fig. 2g). These observations establish that the mesopores of the m-TiO₂ and m-ZrO₂ layers were filled with the perovskite and that the thicknesses of the m-TiO₂, m-ZrO₂ and graphite layers were 0.55, 1.7 and 23 µm, respectively.

Figure 2. Results from the SEM and EDX analyses of the MPLE-PSC device. (a) An SEM image of the unfilled <FTO/m-TiO₂/m-ZrO₂/carbon-graphite> electrode, (b) a magnified image of (a), (c) an EDX line scan of (b), (d) an SEM image of the perovskite filled electrode, (e) a magnified image of (c), (f) an EDX line scan of (e) and (g) an EDX map of the perovskite filled electrode.

Figure 3 presents the results of the J–V measurements using the completed device. Each measurement was performed five times with 3 min intervals under continuous AM1.5G irradiation. Prior to irradiation, the fill factor (FF) associated with the forward scan was particularly low, and significant hysteresis was apparent between the forward and reverse scans. However, during the second set of scans (after 3 min of light irradiation), negligible hysteresis was observed and a standard photovoltaic J–V curve was generated. After 6, 9 and 12 min, the hysteresis was also negligible and the shape of the J–V curves stabilized. The data also indicate that both \( V_{oc} \) and FF were increased as the light irradiation time span was prolonged. For light exposure times of 0, 3, 6, 9 and 12 min, the average \( V_{oc} \) values for the forward and reverse scans were 0.778, 0.866, 0.886, 0.894 and 0.898 V while the FF values were 0.297, 0.626, 0.637, 0.646 and 0.646, respectively. The average PCE values for the forward and reverse scans after these same intervals were 4.52, 10.86, 11.35, 11.59 and 11.67%, respectively. The slight improvement in device performance on going from 3 to 12 min is attributed to enhanced open-circuit photovoltage (\( V_{oc} \)) and FF values. Therefore, to obtain stable J–V data for this architecture, the device had to be irradiated for a sufficient time interval prior to measurements.

Figure 4 shows a schematic diagram of the IPCE measurement process. This measurement was performed using a monochromatic
light source, a mask with an opening of 0.01 cm² and a measurement cell that was in contact with the mask without a gap. As noted above, the IPCE process is a powerful tool for evaluating and comparing the photo-current characteristics of solar cells, and a theoretical value of $J_{sc}$ can be calculated from the IPCE spectrum.\textsuperscript{18}

Figure 5a provides the results of an IPCE analysis of the MPLE-PSC immediately after the activation shown in Fig. 3. Figure 5b summarizes the improvements in the $J_{sc}$ values calculated from the integration of IPCE spectra multiplied with AM1.5G radiation data. A low quantum efficiency (QE) was observed without additional light-soaking (that is, at 0 min). Specially, this QE was lower in the longer wavelengths region (450 to 750 nm). Just after the light activation of the MPLE-PSCs (Fig. 3), the $J_{sc}$ value determined from the $J-V$ data was 20.53 mA cm$^{-2}$. In contrast, the $J_{sc}$ calculated from the IPCE spectra was just 9.21 mA cm$^{-2}$. Evidently, although the MPLE-PSC was activated by applying significant irradiation (that is, employing AM1.5G radiation) during the $J-V$ measurements, the less intense irradiation provided by the monochromatic light during IPCE measurements was insufficient for the purpose of activation. Therefore, trials were also performed using light activation during the IPCE measurement process. Following the initial measurement, light-soaking was performed with a white LED from the side of the glass substrate for a span of 3 min (Fig. 4), under open-circuit conditions. Subsequently, the IPCE was slightly improved (Fig. 5a). This procedure involving 3 min of light-soaking and an IPCE measurement was repeated five times. The IPCE spectrum improved after the third measurement, equivalent to a total light-soaking duration of 6 min. Additional light soaking further improved the IPCE data after the fourth measurement (equal to a total light-soaking time of 9 min), although no further changes were observed in the case of the fifth measurement after a total light-soaking time of 12 min. Therefore, it appears that the MPLE-PSCs was fully activated by 9 min of light-soaking and stabilized thereafter.

The increase in the IPCE values on the long wavelength side of the spectrum (450 to 750 nm) is attributed to the difference of optical absorption coefficient of perovskite crystal. The initial IPCE values were higher on the short wavelength side and lower on the long wavelength side because the optical absorption coefficient for the perovskite crystal was high on the former and low on the latter. Originally, the light absorption coefficient was high on the short wavelength side and the rate of increase of the IPCE value was greater on the long wavelength side where the light absorption coefficient was lower. For these reasons, the short wavelength side was activated more rapidly while the long wavelength side required a longer duration to be activated. Negligible changes were observed in the IPCE values around 350–360 nm because electricity was generated via the adsorption of UV light in the range of 350–360 nm by the TiO$_2$ regardless of the presence of the perovskite.

As can be seen in Figs. 5b and 5c, the calculated $J_{sc}$ values were 9.21, 10.54, 16.36, 19.28 and 19.25 mA cm$^{-2}$ at light-soaking times of 0, 3, 6, 9 and 12 min, respectively. In addition, the averages

![Figure 4](image-url)  
**Figure 4.** A schematic diagram of the IPCE measurement process with light-soaking using an LED.

![Figure 5](image-url)  
**Figure 5.** IPCE spectra obtained after light soaking for 0–12 min. (a) The IPCE spectra, (b) the integrated $J_{sc}$ values obtained with an AM1.5G spectrum and (c) the transition of $J_{sc}$ values during the light soaking produced on the basis of $J-V$ and IPCE measurements.
values for the forward and reverse scans based on the $J-V$ data (Fig. 5c) were 19.53, 20.02, 20.10, 20.53 and 20.09 mA cm$^{-2}$ at these same time intervals. These data confirm that the change in $J_{sc}$ induced by light-soaking was different when using the IPCE and the $J-V$ methods. Finally, the fully activated $J_{sc}$ values were determined to be 19.28 and 20.53 mA cm$^{-2}$ based on the IPCE data and the $J-V$ curve, respectively, giving a difference of 1.25 mA cm$^{-2}$. This difference could possibly be due to leakage of scattered light from the glass substrate to the exterior of the device without absorption by the photoactive area (Fig. 4).

This study demonstrated the stabilization of experimental data resulting from a light-soaking effect during both $J-V$ and IPCE measurements of MPLE-PSCs based on (5-AVA)$_2$(MA)$_2$PbI$_4$. In the case of the $J-V$ analyses, the extent of hysteresis was reduced via this effect, and both $V_{oc}$ and $FF$ were improved with increases in the light-soaking time. During the IPCE measurements, irradiation was supplied by an LED from the side of the glass substrate to explain the purpose of QE activation. The resulting difference of approximately 1 mA cm$^{-2}$ between the $J_{sc}$ values obtained from the $J-V$ and IPCE methods is considered to be acceptable considering the degree of light leakage during the IPCE measurements. The simulated solar radiation light source employed during this work generated a more parallel light flux than the monochromatic light generated by a quartz fiber during the IPCE measurements, and the consequent light dispersion associated with this fiber could have caused the $J_{sc}$ discrepancy of 1.25 mA cm$^{-2}$. Therefore, using parallel monochromatic light may minimize the difference in $J_{sc}$ between these two different methods. The cause of the performance stabilization obtained from this immersion effect in the present trials with 5-AVA-MAPbI$_4$-based MPLE-PSCs is currently under investigation.

Although the associated mechanism remains unclear, the light-activation of MPLE-PSCs is an important research topic because it will allow an evaluation of the stabilized maximum performance of these promising PV devices. Such research will be assisted by obtaining accurate $J-V$ and IPCE data.

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