

Characterization of Perovskite Solar Cell with Fe³⁺ Doped TiO₂ Layer

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Recently, single-crystal silicon solar cells have achieved efficiencies of 25%. However, their production is energy-demanding and relatively expensive. Therefore, photovoltaic cells based on organic–inorganic hybrid perovskites have attracted considerable attention owing to their high conversion efficiencies, simple preparation, and potentially low production costs. In this study, we investigated the effect of Fe³⁺ doped into a compact and mesoporous layer of TiO₂ on the efficiency of the resultant perovskite solar cell (PSC). The efficiencies achieved for PSCs with Fe³⁺/TiO₂ were higher than those for cells with pure TiO₂ nanoparticles. The samples were characterized by X-ray diffraction, scanning electron microscopy, ultraviolet-visible spectroscopy, and current-voltage measurements. The photoelectrode PSC exhibited a high light-to-electric-energy conversion efficiency under a simulated solar light irradiation of 100 mW/cm² (AM 1.5). Our results suggest that Fe³⁺ nanoparticles in the TiO₂ layer may contribute to the performance improvement of the PSC.

Keywords: Perovskite Solar Cell, Mesoporous TiO₂, Fe³⁺-Doped TiO₂.

1. INTRODUCTION

Over the past three decades, particularly over the past few years, the development of renewable energy technologies has attracted significant attention to overcome the issues of environmental pollution and global warming [1]. Recently, a new type of solar cell based on an organic–inorganic halide perovskite material has been developed. Perovskite solar cells (PSCs) were reported in 2009; their efficiencies rapidly increased, from 3.8% to 22% [2–5]. PSCs have many advantages owing to the use of simple processing technology and stable materials. Perovskites materials have a general formula of ABX₃, where A is generally methyl ammonium (CH₃NH₃⁺ (MA)), B is a metal ion, such as Pb or Sn, and X represents a halogen ion, such as I, Cl, or Br. A distinct advantage of lead-based perovskites (i.e., MAPbX₃) is that their band gaps can be easily tuned, from 1.2 to 2.3 eV, by varying the compositions of the cations and anions [6, 7]. In addition, the low density of interface states, high transparency, and chemical stability also contribute to the performances of the solar cells. In order to meet these requirements, TiO₂ is often used as the *n*-type layer in planar PSCs. The *p–i–n* architecture has been widely employed in both earlier mesoporous TiO₂-based devices and later planar heterojunction

devices, providing both hole- and electron-transport layers (ETL) with high performances [8–10]. In these PSCs, the ETL transmits electrons and blocks holes. The conduction band of TiO₂ used in many PSCs coincides well with the conduction band of the perovskite active layer. In addition, TiO₂ has excellent stability and conductivity and can be obtained by various deposition methods at a low cost. In this regard, in many PSCs, TiO₂ is widely used as the ETL owing to its excellent electronic properties [11–15]. However, TiO₂ traps electrons in oxygen vacancies and Ti lattice sites, which leads to recombination, which in turn reduces the efficiency of the device. Therefore, the generation of oxygen vacancies and reduction in the number of defects of TiO₂ facilitate the development of stable PSCs. Many studies have shown that the efficiency can be effectively improved by reducing the recombination of TiO₂ by doping appropriate metal ions.

In this study, we investigated the effects of Fe³⁺ ions on the efficiencies of PSCs by doping them in a compact and mesoporous layer of TiO₂. Fe³⁺ was dissolved in ethanol and mixed with a TiO₂ solution in various ratios. The metal ion can effectively enhance the electron transfer of TiO₂. Fe³⁺ with an atomic radius similar to that of Ti⁴⁺ can replace Ti cations, and Fe³⁺ doped TiO₂ is considered to be a better candidate for photoelectric performance of PSCs.

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Upon the doping with Fe³⁺, the conductivity increased and the recombination decreased, which increased the efficiency of the PSC.

2. EXPERIMENTAL METHODS

Lead iodide (PbI₂, 99.999%), Fe(NO₃)₃ · 9H₂O, 4-tert-butylpyridine (4-tBP), methyl ammonium iodide (CH₃NH₃I), lithium bis(trifluoromethanesulfonyl)imide (Li-TFSI), acetonitrile (ACN, 99.8%), 2,2',7,7'-tetrakis-(N,N-di-p-methoxyphenyl-amine)-9,9'-spirobifluorene (spiro-MeOTAD), N,N-dimethylformamide (DMF, 99.8%), dimethylsulfoxide (DMSO), and chlorobenzene (CB) were purchased from Sigma-Aldrich, whereas a TiO₂ paste (18 NR-T) and CH₃NH₃I (MAI) were obtained from Dyesol. All of the materials were used as received without purification.

Fluorine-doped tin oxide (FTO)-coated glass substrates were cleaned using an ultrasonic bath with acetone, isopropanol, and deionized water and were then dried under nitrogen gas. A 0.15-M TiO₂ precursor solution was prepared from titanium diisopropoxide bis (acetylacetonate) in ethanol to prepare the TiO₂ compact layer solution. 0.1 g of Fe(NO₃)₃ · 9H₂O was weighed and dissolved in 2 mL of ethanol and then stirred for 5 h.

An Fe(NO₃)₃ solution was then added to TiO₂ at various molar ratios and stirred for 5 h. The prepared solution was spin-coated on the FTO substrate at 3000 rpm for 30 s, heat-treated at 450 °C for 15 min, and then cooled at room temperature. The TiO₂ paste (mass ratio with respect to ethanol: 1:4) was prepared by adding dissolved Fe(NO₃)₃ at various molar ratios and stirring for 5 h. A spin coating was then carried out at 6000 rpm for 30 s followed by a heat treatment at 450 °C for 15 min and subsequent cooling to room temperature.

Subsequently, the TiO₂ paste without Fe(NO₃)₃ was spin-coated at 6000 rpm for 30 s, heat-treated at 450 °C for 15 min, and then cooled at room temperature. The Fe(NO₃)₃ solution was then added to TiO₂ at various molar ratios and stirred for 5 h.

The perovskite layers were deposited by spin coating at 3000 rpm for 30 s with 1 mol of PbI₂ in DMF/DMSO (10:1) and then DMF was evaporated with vacuum. The solution was prepared by dissolving 20 mg of MAI in 1 mL of IPA and coated 10 times (10 μL) at 4000 rpm. The heat treatment was performed at 150 °C for 10 min and 120 °C for 30 min in an N₂-gas-filled box, followed by cooling to room temperature.

The hole-transport material solution was prepared with 72.3 mg of spiro-OMeTAD, 28.8 μL of 4-tBP, and 17.5 μL of a Li-TFSI solution (520 mg of Li-TFSI in 1 mL of acetonitrile) dissolved in 1 mL of chlorobenzene. This solution was spin-coated onto the perovskite layer at 4000 rpm for 30 s. Finally, a 60-nm-thick Au layer was deposited as the back contact by thermal evaporation.

The performance of the fabricated PSC was evaluated using a current-voltage solar simulator (K3000, McScience) and solar cell quantum efficiency test system (K3100, McScience). The methylammonium lead halide structure was analyzed by X-ray diffraction (XRD) spectroscopy (D/Max-2200 diffractometer, Rigaku), field-emission scanning electron microscopy (SEM, S-4700, Hitachi), and ultraviolet-visible spectrometry (UV-Vis 8453, Agilent). Electrochemical impedance spectroscopy (EIS) measurements were carried out under dark conditions using an electrochemical workstation (ZAHENR, Germany).

3. RESULTS AND DISCUSSION

An XRD measurement is performed to reveal the lattice structure. Figure 1(a) shows the XRD pattern of Fe³⁺ undoped TiO₂. As shown in Figure 1(b), the Fe³⁺-doped TiO₂ layer consists of the anatase phase, as the Fe³⁺ ion radius is very similar to that of Ti⁴⁺ and thus the Fe³⁺ dopant cannot change the lattice of TiO₂ [16–18]. Figure 1(c) shows the XRD pattern of TiO₂/CH₃NH₃PbI₃ with peaks at 13.8, 19.7, 23.1, 24.2, 28.1, 31.5, 40.3, and 42.8° attributed to the (110), (112), (211), (222), (224), and (314) lattice planes, respectively. These results demonstrate the tetragonal structure of the perovskite. The XRD patterns of our samples are similar to those of the mesoporous perovskite layers recorded previously [19].

Figures 2(a) and (b) show that the TiO₂ layer is homogeneous and there is no difference after doping of Fe³⁺. Figure 2(c) shows that the coated TiO₂ has a thickness of about 400–500 nm. The cross-sectional SEM image of the PSC in Figure 2(d) shows that perovskite thin films with large crystal grains were produced.

Figure 3 shows the UV-vis absorbances of the samples of (a) CP TiO₂/MP TiO₂, (b) 1 mol% Fe³⁺-CP TiO₂/0.5 mol% Fe³⁺-MP TiO₂, (c) 1 mol% Fe³⁺-CP TiO₂/1 mol% Fe³⁺-MP TiO₂, (d) 1 mol% Fe³⁺-CP TiO₂/2 mol%

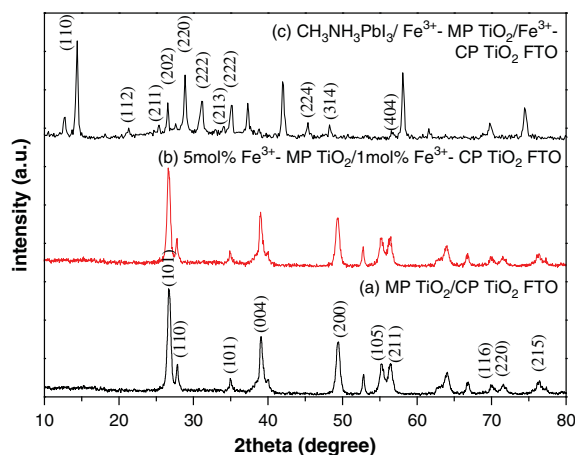


Figure 1. XRD patterns of MAPbI₃, Fe³⁺-TiO₂ and TiO₂.

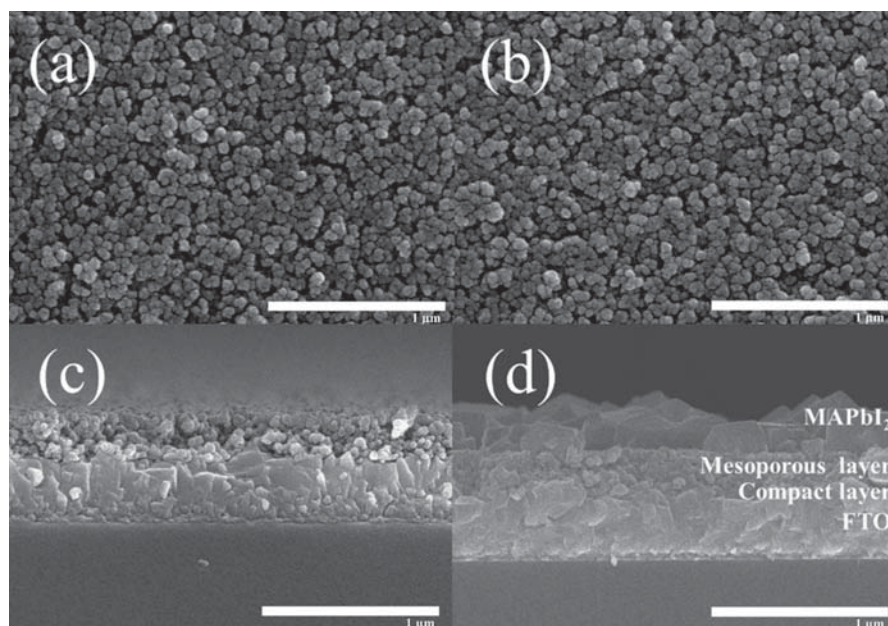


Figure 2. SEM image of (a) TiO₂/FTO (b) Fe³⁺-TiO₂/FTO, (c) SEM image of its cross section, and (d) cross-sectional SEM image of MAPbI₃/Fe³⁺-TiO₂/FTO.

Fe³⁺-MP TiO₂, and (e) 1 mol% Fe³⁺-CP TiO₂/5 mol% Fe³⁺-MP TiO₂. The absorbance increases with the Fe³⁺ doping concentration. This has been attributed to the formation of an Fe-dopant energy level within the band gap of TiO₂ [20]. The Fe³⁺ doping of the TiO₂ layer does not significantly affect the MAPbI₃ film in the visible range [21].

The *J*-*V* curve in Figure 4 shows that the photovoltaic properties of the PSCs using Fe³⁺-doped TiO₂ were improved. The corresponding photovoltaic properties are

summarized in Table I. The best performance is obtained for the 1-mol% mixed sample. Oxygen vacancies or Ti lattice sites are converted to Ti³⁺ by stoichiometric defects in the TiO₂ lattice [20]. Therefore, the Ti³⁺ lattice defects can induce low energy levels and the conduction band can trap electrons and reduce the electron transport efficiency, leading to recombination. The increase in open-circuit voltage (*V*_{oc}) is worth discussing. *V*_{oc} of a perovskite solar cell is determined by the characteristics of each material [22, 23]. When TiO₂ is doped with metal cations, the TiO₂ energy level is changed and the *V*_{oc} value is

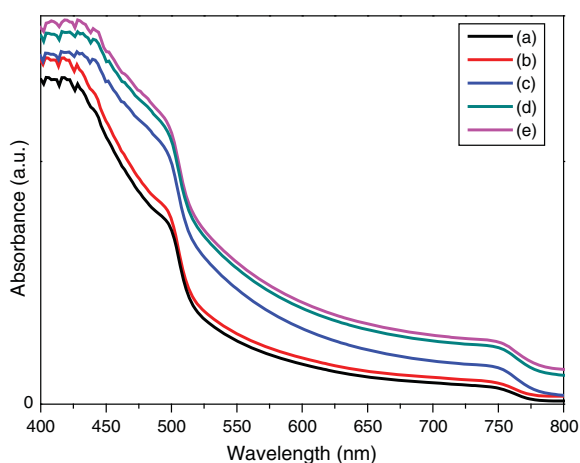


Figure 3. UV-vis absorbances of (a) CP TiO₂/MP TiO₂, (b) 1 mol% Fe³⁺-CP TiO₂/0.5 mol% Fe³⁺-MP TiO₂, (c) 1 mol% Fe³⁺-CP TiO₂/1 mol% Fe³⁺-MP TiO₂, (d) 1 mol% Fe³⁺-CP TiO₂/2 mol% Fe³⁺-MP TiO₂, and (e) 1 mol% Fe³⁺-CP TiO₂/5 mol% Fe³⁺-MP TiO₂.

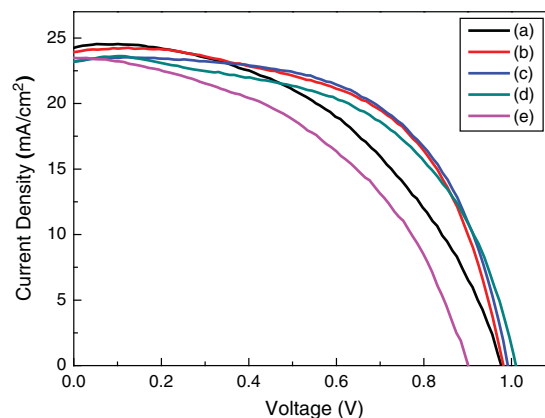


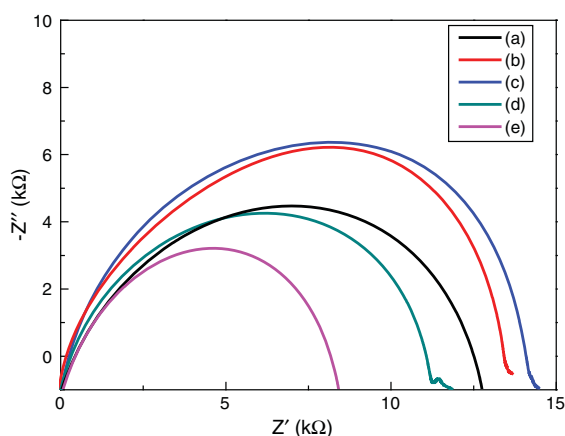
Figure 4. *J*-*V* curves of the Fe³⁺-TiO₂-based PSCs of (a) CP TiO₂/MP TiO₂, (b) 1 mol% Fe³⁺-CP TiO₂/0.5 mol% Fe³⁺-MP TiO₂, (c) 1 mol% Fe³⁺-CP TiO₂/1 mol% Fe³⁺-MP TiO₂, (d) 1 mol% Fe³⁺-CP TiO₂/2 mol% Fe³⁺-MP TiO₂, and (e) 1 mol% Fe³⁺-CP TiO₂/5 mol% Fe³⁺-MP TiO₂.

Table 1. Current density–voltage characteristics of best performing perovskite solar cell.

Concentration	V_{oc} (V)	J_{sc} , (mA/cm ²)	FF (%)	η (%)	R_s (ohm)	R_{sh} (ohm)
0	0.978	24.25	48.37	11.47	255	6706
0.5	0.981	23.92	58.45	13.72	158	8890
1.0	0.992	23.18	60.19	13.84	167	32826
2.0	1.010	23.19	55.69	13.04	195	5330
5.0	0.901	23.47	46.36	9.80	238	3076

increased [23]. If Fe³⁺ ions substitute Ti⁴⁺ ions at Ti lattice sites in TiO₂, the electronic energy level of the oxide film is elevated, which leads to an increase in V_{oc} . In addition, V_{oc} is increased when the electron recombination is reduced. V_{oc} of the PSCs increases from 0.978 V to the 1.01 V, suggesting less energy loss of electrons [24]. Fe³⁺ doped TiO₂ can expect enhanced photovoltaic performance of PSC. The decrease in V_{oc} is due to the fact that introduction of Fe ion produces defects in the oxide film, which causes the recombination of photo-induced holes and electrons [25]. The series resistance (R_s) and the shunt resistance (R_{sh}) are defined by the diode equation. Reductions in series show the mobility of electrons in the TiO₂ layer. The increase in R_{sh} is due to the rapid extraction of electrons and less trap-assisted recombination [26–29]. Rapid extraction of electrons reduces the frequency at which electrons recombine with holes. A low R_s and high R_{sh} can lead to a high fill factor (FF) [30].

Figure 5 shows an EIS characterizing the solar cell internal resistance. The PSC is considered as a leakage capacitor in the absence of light [31]. The semicircle is related to the charge recombination at the CP TiO₂/MP TiO₂/CH₃NH₃PbI₃/spiro-OMeTAD interface. When Fe³⁺ is doped, the V_{oc} increases and the electron energy loss

**Figure 5.** EIS used to characterize the PSC internal resistances: (a) CP TiO₂/MP TiO₂, (b) 1 mol% Fe³⁺-CP TiO₂/0.5 mol% Fe³⁺-MP TiO₂, (c) 1 mol% Fe³⁺-CP TiO₂/1 mol% Fe³⁺-MP TiO₂, (d) 1 mol% Fe³⁺-CP TiO₂/2 mol% Fe³⁺-MP TiO₂, and (e) 1 mol% Fe³⁺-CP TiO₂/5 mol% Fe³⁺-MP TiO₂.

decreases. Also, the increase in R_{sh} causes a decrease in electron recombination. According to the equivalent circuit description of the solar cell, R_{sh} is closely related to charge recombination at the interface inside the device. The wide semicircle shows that the electron–hole recombination rate is low [32]. This is beneficial for the charge transfer from the perovskite to TiO₂ [33].

4. CONCLUSION

We fabricated PSCs based on MAPbI₃ using TiO₂ and different Fe³⁺ concentrations. The doped Fe³⁺ did change the Ti lattice. The absorbance increased with the concentration. The TiO₂ layer doped at a concentration of 1 mol% had a homogeneous and uniform surface and reduced electron recombination, leading to the improved electrical conductivity. The PSC exhibited a short-circuit current density (J_{sc}) of 23.18 mA/cm², V_{oc} of 0.992 V, FF of 60.19%, and efficiency (η) of 13.84%. Therefore, the use of the Fe³⁺-doped TiO₂ layer is a promising approach for fabrication of high-performance PSCs.

Acknowledgments: This work was supported by the Korea Institute of Energy Technology Evaluation and Planning (KETEP) and the Ministry of Trade, Industry and Energy (MOTIE) of the Republic of Korea (No. 20194030202290). This research was supported by Korea Electric Power Corporation. (Grant number: R18XA02).

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Received: 15 December 2018. Accepted: 26 March 2019.