Fabrication and characterization of potassium- and formamidinium-added perovskite solar cells

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Perovskite solutions with potassium (K) and formamidinium (FA) iodides added were used to fabricate perovskite solar cells. Since the lattice constants increased with the addition of FA, the substitution by FA of the CH₃NH₃ (MA) site of the perovskite crystal was confirmed. In addition, conversion efficiencies were improved for devices with K and FA added compared with standard devices. The presence of K in the perovskite solution promoted formation of highly (002)-oriented crystals, which decreased the lattice strain of perovskite crystals. Filling the MA defect sites with K and FA can prevent the recombination of electrons and holes and improve the photovoltaic characteristics.

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Key-words : Perovskite, Solar cell, Potassium, Formamidinium

1. Introduction

Although the most common solar cells are currently silicon solar cells, use of silicon solar cells as power-generation devices remains expensive. Recently developed perovskite solar cells using CH₃NH₃PbI₃ (MAPbI₃) compounds that can be fabricated relatively easily¹⁻⁶ are expected to be candidates for next-generation solar cells.⁷⁻¹³ Perovskite CH₃NH₃PbI₃ crystals make it possible to control electronic states by doping the cation and anion positions and with selected elements and molecules.¹⁴⁻¹⁹ Adding alkali metal ions such as Cs⁺ and Rb⁺ to perovskite precursor solutions induces nucleation processes and crystallization, leading to increased grain sizes and reduced defect densities, and these alkali metal processes have been reported to provide high conversion efficiencies and stability.²⁰⁻²³ Conversion efficiency can also be improved by using K, which offers lower cost and greater abundance than Cs and Rb. It is expected that the semiconductor characteristics of perovskite films can be adjusted by appropriate K⁺ doping.²⁴⁻³⁰ A tolerance factor (t-factor) calculated from ionic radii that can predict the stability of perovskite structures³¹⁻³⁹ shows that doping with K⁺ alone reduces the structural stability of perovskite compounds,³⁰ and other cations with larger ionic radii may consequently be necessary to form stable perovskite structures.

The purpose of this study was to fabricate and characterize perovskite solar cells in which potassium iodide (KI) and formamidinium [HC(NH₂)₂, FA] iodide were added to standard MAPbI₃. The co-addition of K and FA to MAPbI₃ was expected to stabilize the perovskite structure. The annealing temperatures for the formation of perovskite layers with alkali metal ions added are commonly ~100 °C, and this low-temperature annealing might cause instability. Since high-temperature annealing could be effective for the formation of more stable perovskites,⁴⁰⁻⁴¹ hot air-blowing and higher temperature annealing in ambient air were applied in the present work. In addition to KI, the amount of FA added to the perovskites was also evaluated. Since HC(NH₂)₂PbI₃ (FAPbI₃) including an FA⁺ cation has a smaller energy gap (Eₜ) of 1.48 eV than MAPbI₃ (1.55 eV), the wavelength range for photovoltaic conversion could be expanded, and improvements in short-circuit current density and conversion efficiency were expected. The effects of KI and FA co-addition to perovskite crystals on their photovoltaic properties were investigated using light-induced current density–voltage (J–V) measurements, external quantum efficiency (EQE), X-ray diffraction (XRD), optical microscopy, and scanning electron microscopy (SEM).

2. Experimental procedures

A schematic illustration showing the fabrication processes used to fabricate photovoltaic cells is shown in Fig. 1. All processes were performed under conditions of ~27 °C temperatures and ~40 % humidity in ambient air. F-doped tin oxide (FTO, Nippon Sheet Glass Company, ~10 Ω/□) substrates were cleaned in an ultrasonic bath with acetone and methanol, and dried under nitrogen gas. The FTO substrates were treated with an ultraviolet ozone
Pure Chemical Industries) at 60 °C. Standard MAPbI3 precursor solutions (0.15 and 0.30 M) were prepared from titanium diisopropoxide bis(acetyl acetonate) (Sigma Aldrich) and 1-butanol (Wako Pure Chemical Industries). Both TiO2 precursor solutions were spin-coated onto the FTO substrate at 3000 rpm for 30 s and annealed at 125 °C for 5 min, and the 0.30 M precursor was spin-coated twice to form a uniform layer. After that, the FTO substrate was annealed at 550 °C for 30 min to form a compact TiO2 layer. A mesoporous TiO2 precursor solution was spin-coated onto the mesoporous TiO2 layer at 5000 rpm for 30 s using TiO2 paste. The TiO2 paste was prepared using TiO2 powder (Aerosil P-25, 200 mg) and poly(ethylene glycol) layer. A mesoporous TiO2 precursor solution was spin-cleaned (Asumi Giken ASM401N) for 15 min. TiO2 was annealed at 550 °C for 30 min to form a compact TiO2 layer. A mesoporous TiO2 precursor solution was spin-coated onto the mesoporous TiO2 layer at 5000 rpm for 30 s using TiO2 paste. The TiO2 paste was prepared using TiO2 powder (Aerosil P-25, 200 mg) and poly(ethylene glycol) (Nacalai Tesque PEG #20000, 20 mg) in distilled water (0.5 mL). This solution was mixed with acetylacetone (Wako Pure Chemical Industries, 20 μL) and the Triton X-100 surfactant (Sigma Aldrich, 10 μL) for 30 min and then was allowed to stand for ~24 h to suppress bubble formation in the solution. The FTO substrates with the TiO2 layers were annealed at 550 °C for 30 min to form mesoporous TiO2 layers.

The perovskite compounds were prepared by mixing CH3NH3I (Showa Chemical), HC(NH2)2I (Tokyo Chemical Industries), PbCl2 (Sigma Aldrich), and KI (Wako Pure Chemical Industries) with molar concentrations of MAI and PbCl2 of 2.4 and 0.8 M, respectively, and were prepared, as summarized in Table 1. As the FA composition increased, the J-factor increased to 1, indicating that the crystal distortion in the perovskite structure was reduced by the addition of FA. Solutions containing the perovskite precursors were spin-coated onto the mesoporous TiO2 layers at 2000 rpm for 60 s using the hot air-blowing method.41) The temperature of the FTO substrates during hot air-blowing was measured at 90 °C. The cells were then annealed at 160 °C for 10 min in ambient air.

A hole transport layer (HTL) was formed by spin-coating at 4000 rpm for 30 s. The HTL precursor solution was prepared by adding 2,2’,7,7’-tetrais(N,N-di-p-methoxyphenylamine)-9,9’-spirobifluorene (spiro-OMeTAD, Sigma Aldrich 36.1 mg) to chlorobenzene (0.5 mL; Wako Pure Chemical Industries) and stirring the solution for 24 h. A solution of lithium bis(trifluoromethylsulfonyl)imide (Li-TFSI, Tokyo Chemical Industry, 260 mg) in acetonitrile (Nacalai Tesque, 0.5 mL) was also prepared by stirring for 24 h. The former spiro-OMeTAD solution with 4-tert-butylpyridine (Sigma Aldrich, 14.4 μL) was mixed with the latter Li-TFSI solution (8.8 μL) for 30 min at 70 °C, and cooled to ambient temperature. Finally, a gold (Au) top-electrode was formed by vacuum evaporation.

The J–V characteristics (Keysight, B2901A) of the photovoltaic cells were measured when illuminated with a solar simulator (San-ei Electric, XES-301S) at 100 mW cm⁻² with a 1.5 air mass. The solar cells were illuminated through the side of the FTO substrates, and the exposed area was 0.080 cm². The EQEs of the solar cells were also measured using an incident photon-to-electron conversion efficiency measurement system (Eni Technologie, QE-R). The microstructures of the perovskite layers were investigated with an X-ray diffractometer (Bruker, D2 PHASER), while the surface morphologies of the perovskite layers were examined using an optical microscope (Nikon, Eclipse E600) and a scanning electron microscope (Jeol, JSM-6010PLUS/LA) equipped with energy dispersive X-ray spectroscopy (EDS).

3. Results and discussion

The J–V characteristics of the photovoltaic devices containing K and FA are shown in Fig. 2. The photovoltaic parameters of these devices are summarized in Table 2, where $J_{SC}$ is the short-circuit current density, $V_{OC}$ is the open-circuit voltage, FF is the fill factor, $R_s$ is the series resistance, $R_{sh}$ is the shunt resistance, $\eta$ is the conversion efficiency, and $\eta_{ave}$ is the average efficiency of three cells. The device containing the standard MAPbI3 perovskite had an $\eta$ of 1.38 %, while the devices containing the K- and FA-doped perovskites had $\eta$ efficiencies in the range of 7–13 %. The highest $\eta$ of 13.53 % was obtained for the device containing the MA0.64FA0.31K0.05PbI3 perovskite.

Table 1. Compositions and calculated J-factors of the present perovskite compounds

| Perovskite          | K (%) | FA (%) | J-factor |
|---------------------|-------|--------|----------|
| CH3NH3PbI3          | 0     | 0      | 0.912    |
| CH3NH3[HC(NH2)2]0.86K0.14PbI3 | 5  | 10     | 0.914    |
| CH3NH3[HC(NH2)2]0.86K0.14PbI3 | 5  | 20     | 0.921    |
| CH3NH3[HC(NH2)2]0.86K0.14PbI3 | 5  | 31     | 0.929    |
| CH3NH3[HC(NH2)2]0.86K0.14PbI3 | 5  | 47     | 0.941    |
The JSC and VOC of the devices containing the K- and FA-doped perovskites were higher than those of the standard device, resulting in improved conversion efficiencies.

EQE spectra of the photovoltaic devices are shown in Fig. 3. The estimated energy gaps in the perovskite crystals, calculated by linear fitting using band gap calculator software (Enlitech), are summarized in Table 3. The energy gaps in the perovskite crystals decreased from 1.55 to 1.52 eV with the addition of K and FA. The EQEs of the devices containing the K- and FA-doped perovskites were higher than that of the standard device between 470 and 815 nm, which resulted in an increase in JSC.

Optical microscopy images of the perovskite compounds in the devices are shown in Fig. 4. Gaps of a few μm between the perovskite particles are observed for the standard device without K and FA, as shown in Fig. 4(a). When K and FA were added, the gaps between the perovskite particles were reduced compared with those in the standard perovskite, as shown in Figs. 4(b) and 4(c). The perovskite particles appear to be densely packed, which accounts for the increase in the conversion efficiency of the devices with MA0.85FA0.10K0.05PbI3 [Fig. 4(b)] and MA0.64FA0.31K0.05PbI3 [Fig. 4(c)] added. Some gaps are observed between the perovskite particles in the 47% FAI-added device, as shown in Fig. 4(d), and this might decrease the JSC of the cells.

Table 2. Photovoltaic parameters of the present perovskite solar cells

| Perovskite | JSC (mA cm⁻²) | VOC (V) | FF | Rp (Ω cm²) | Ra (Ω cm²) | η (%) | ηave (%) |
|------------|---------------|--------|----|------------|-----------|-------|---------|
| 0 0        | 10.5          | 0.417  | 0.210 | 33.4       | 34.3      | 1.38  | 1.28    |
| 5 0        | 9.2           | 0.307  | 0.254 | 32.6       | 35.0      | 0.72  | 0.59    |
| 5 10       | 16.5          | 0.792  | 0.562 | 6.54       | 253       | 7.35  | 7.04    |
| 5 20       | 23.5          | 0.829  | 0.643 | 5.75       | 543       | 12.52 | 10.59   |
| 5 31       | 21.8          | 0.902  | 0.689 | 4.68       | 687       | 13.53 | 12.42   |
| 5 47       | 20.0          | 0.903  | 0.686 | 5.32       | 669       | 12.38 | 11.76   |

Fig. 2. J–V characteristics of the present perovskite solar cells.

The JSC and VOC of the devices containing the K- and FA-doped perovskites were higher than those of the standard device, with resulting improved conversion efficiencies.

EQE spectra of the photovoltaic devices are shown in Fig. 3. The estimated energy gaps in the perovskite crystals, calculated by linear fitting using band gap calculator software (Enlitech), are summarized in Table 3. The energy gaps in the perovskite crystals decreased from 1.55 to 1.52 eV with the addition of K and FA. The EQEs of the devices containing the K- and FA-doped perovskites were higher than that of the standard device between 470 and 815 nm, which resulted in an increase in JSC.

Fig. 3. EQE of the present perovskite solar cells.

Table 3. Energy gaps and lattice parameters of the perovskite crystals. P: unit cell volume. Z: number of chemical units in the unit cell. D: crystallite size. I₀₀₂/I₁₁₄: ratios of (002) diffraction intensities (I₀₀₂) to (114) diffraction intensities (I₁₁₄).

| Perovskite | E₉ (eV) | Lattice constant | V (Å³) | Z (Å³) | I₀₀₂/I₁₁₄ |
|------------|---------|-----------------|--------|--------|-----------|
| 0 0        | 1.55    | 8.848           | 12.54  | 981.7  | 4 245.4   |
| 5 0        | 1.54    | 8.871(1)        | 12.55(6)| 983.1  | 4 245.8   |
| 5 10       | 1.53    | 8.872           | 12.51  | 984.7  | 4 246.2   |
| 5 20       | 1.52    | 8.873(3)        | 12.57(4)| 990.3  | 4 247.5   |
| 5 31       | 1.53    | 8.888           | 12.60  | 995.4  | 4 248.8   |
| 5 47       | 1.53    | 8.899(1)        | 12.61(2)| 998.2  | 4 249.5   |

Fig. 4. Optical microscope images of the perovskite solar cells that contained K and FA-added perovskites. (a) Standard MAPbI₃, (b) MA₀.₈₅FA₀.₁₀K₀.₀₅PbI₃, (c) MA₀.₆₄FA₀.₃₁K₀.₀₅PbI₃, and (d) MA₀.₄₅FA₀.₄₇K₀.₀₅PbI₃.

Figure 5(a) shows an SEM image of the MA₀.₆₄FA₀.₃₁-K₀.₀₅PbI₃ cell containing 5% KI. The average particle size is ~5μm, and the particles are interconnected. These
connected surface structures might improve the surface coverage and, therefore, the photovoltaic properties. Figures 5(b)–5(d) show EDS elemental mapping images of Pb, I, and K, respectively. The EDS indicates that Pb, I, and K appear to be dispersed throughout the matrix.

XRD patterns of standard MAPbI₃ cells and cells with KI and FAI added, indexed with tetragonal symmetry, are shown in Fig. 6(a). The devices with K and FA exhibited highly (002)-oriented perovskite crystals, and the 002 and 004 peak intensities increased significantly with the addition of KI and FAI. The high intensity of the 002 reflection indicates that the flat surfaces of the particles in Fig. 5(a) correspond to the (002) plane of the tetragonal perovskite. The (114), (006), and (330) reflections cannot be observed in Fig. 6(a) due to their very weak intensities.

The intensified XRD patterns of the (114), (006), and (330) peaks are shown in Figs. 6(b) and 6(c). Figure 6(c) indicates peak shifts of 006 reflections to lower diffraction angles. The measured lattice constants of the perovskite compounds are summarized in Table 3, which shows that the unit cell volumes increased with the addition of KI and FAI compared with that of standard MAPbI₃. X-rays irradiated only the perovskite layers between the Au electrodes of the present solar cell devices, and there should, therefore, be no Au peaks in the present measurement configurations. Since slightly broadened X-rays sometimes irritated the edges of the Au electrodes, diffraction peaks corresponding to Au appeared with various intensities, as observed in Fig. 6(a).

The ratios of the 002 diffraction intensities (I₀₀₂) to the 114 diffraction intensities (I₁₁₄) of the perovskite crystals were calculated as I₀₀₂/I₁₁₄ from the XRD data in Fig. 5 and summarized in Table 3. If the tetragonal MAPbI₃ perovskite particles were randomly oriented, the I₀₀₂/I₁₁₄ value should be 1.83. Most of the I₀₀₂/I₁₁₄ values in the present work were greater than ~200, however, indicating that the (002) planes of the present perovskite particles are extremely preferentially oriented parallel to the FTO substrate compared with randomly oriented perovskite particles. These highly oriented crystals with large crystallite sizes were formed by the hot air-blowing.

Enlarged XRD patterns of 300 reflections of FAI-added cubic perovskite crystals are shown in Fig. 6(d), where the diffraction index of 300 for cubic symmetry corresponds to the 006 and 330 indices for tetragonal symmetry at around 43°. Changes in the lattice constants for the FA-added perovskite are summarized in Table 4. The unit cell volume of the perovskite crystal is related to the presence of defects, which influence device performance. Differences...
between the lattice constants of MAPbI3 in the single crystal and thin films in the present work are likely due to MA defects in the perovskite structures. When the lattice constant of single crystal MAPbI3 is 6.315 Å, the unit cell volume of this perovskite crystal is 251.837 Å³. If the ionic radii of MA, FA, Pb, I and Cl are 2.17, 2.53, 1.19, 2.20 and 1.81 Å, respectively, the sum of the volumes calculated from each ionic radius in the MAPbI3 is 183.668 Å³. The gap ratio is then calculated to be 27.07 %, as listed in Table 4. In the present work, MAPbI3 and all other perovskite crystals were doped with a small amount (4 %) of Cl, and the calculated models also include this Cl. It was assumed that the gap ratio of the Cl-doped perovskite was the same as that of MAPbI3, and the unit cell volume was calculated to be 248.585 Å³. The volume occupancy was then defined as (measured unit cell volume)/ (unit cell volume calculated from the ionic radii), and the result was 98.7 % for the perovskite crystal in the standard cell, as shown in Table 4. This indicates that MA was deficient in the perovskite crystal, and that a small amount of MACl might have vaporized from the perovskite crystal during annealing at 160 °C. If the difference between the unit cell volumes of the previously reported single crystal data and the present experimental data was due to desorption of MACl, the number of MACl defects can be calculated from their ionic radii. Numbers of the Schottky defects (n) are expressed by the following equation: 

\[ n = \frac{N e^{\frac{-E_v}{k_B T}}}{V_{\text{ratio}} / \frac{V_{c} \text{calc}}{V_{c} \text{meas}}} \]

where \( N \) is the number of atoms per volume, \( E_v \) is the activation energy for the formation energy of defects, \( k_B \) is the Boltzmann constant, and \( T \) is the temperature (298 K). Thus, the number for the defects in the MAPbI3(Cl) crystal and the energy for defect formation was calculated as 1.4 \times 10^{20} \text{ cm}^{-3} and 0.087 eV, respectively. Energies for defect generation for MA, I and Pb were calculated as 0.58, 0.84 and 2.31 eV, respectively. The estimated energy for defect generation in the present work is significantly smaller than that for ordinary ionic crystals, indicating that MA may be easily desorbed from microcrystalline thin films during rapid annealing and cooling.

On the other hand, the lattice constant of FA-PbI2 was reported to be 6.362 Å, and the unit cell volume to be 257.502 Å³. These values are larger than those of MAPbI3 due to the larger ionic radius of FA + compared to that of MA +. The gap ratios, calculated on the assumption of a linear relationship with the lattice constant, are summarized in Table 4. The volume occupancies were then calculated, showing an increase of up to 99.9 % for FA 47 % crystal, as shown in Table 4, which indicates that the MA vacancies can be filled with FA.

The EDS mappings of K in Fig. 5(d) show that the K atoms are distributed in both the perovskite grains and grain boundaries, and Fig. 5(d) also indicates some little segregation of K. In the present work, two models were proposed for the role of K atoms. The first model is a passivation effect of disordered regions at the surfaces and boundaries of perovskite grains. There should be some defects at the surfaces or boundaries of grains, as shown in Fig. 8(a), and K atoms with a smaller ionic size would precipitate and cover the disordered regions [Fig. 8(b)], which might compensate the recombination at the defects and lead to improvement of the carrier transport. Changes in the unit cell volumes cannot be described appropriately using this model, however, and other models might be needed to explain the volume changes occurring with the addition of K.

The second model is a K-doping model at the MA site. The unit cell volumes in Tables 3 and 4 were plotted as shown in Fig. 7, and the unit cell volumes of FA-added perovskites without K were compared with those of perovskite with K/FA co-added. For the perovskite without FA, the unit cell volume increased with K addition. This implies that the added K occupied the vacant MA sites to expand the perovskite lattice. This lattice distortion is illustrated schematically in the assumed structural model shown in Fig. 8(c). On the other hand, when K was added to FA-based perovskites, the unit cell volumes decreased. This implies that the filled MA/FA sites were substituted by the added K + with its smaller ionic radius.
resulting in a decrease in the unit cell volumes, as shown in the assumed structural model in Fig. 8(d).

Schematic illustrations of carrier transport for the standard and K/FA-added perovskites are shown in the cross-sectional microstructures in Figs. 8(a) and 8(b), respectively. There are MA defects in the standard MAPbI₃ crystals, as described above, and the electrons and holes were trapped at the MA defects. When K/FA were introduced at the MA defects, carrier trapping was suppressed, which resulted in an improvement of conversion efficiencies.

4. Conclusion

The effects adding KI and FAI to the perovskite precursors on the microstructures and photovoltaic properties of devices containing these materials were investigated. The $J$–$V$ characteristics showed improvement of the device performance achieved by introducing K and FA into the MAPbI₃ perovskite, and the device with KI 5% and FAI 31% added exhibited the highest conversion efficiency of 13.53%. By using the air-blowing method during the process, a high (002)-orientation of the tetragonal perovskite crystal was formed, and the unit cell volumes increased with the amount of FA added. The addition of K also reduced the number of MA/FA cation defects in the perovskite crystals, resulting in a decrease in the lattice strain and trapping sites of electrons and holes. This study showed that the addition of K/FA to MAPbI₃ perovskites using the air-blowing method is an effective method of fabricating photovoltaic devices with improved properties.

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