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Cite as: AIP Advances 8, 105122 (2018); https://doi.org/10.1063/1.5054347
Submitted: 31 August 2018 . Accepted: 25 September 2018 . Published Online: 18 October 2018

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Flexible and transparent IWO films prepared by plasma arc ion plating for flexible perovskite solar cells

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(Received 31 August 2018; accepted 25 September 2018; published online 18 October 2018)

We investigated flexible W-doped In$_2$O$_3$ (IWO) electrodes prepared by arc plasma ion plating (APIP) as a substitute for sputtered amorphous ITO electrodes in flexible perovskite solar cells (FPSCs). In spite of the room temperature ion plating process, the APIP-grown IWO film showed a low sheet resistance of 37.14 Ohm/square, a high optical transmittance of 96.0%, high near IR transmittance, and a small bending radius of 5 mm. In addition, the IWO film shows an atomically smooth surface with a root mean square roughness of 0.83 nm due to the absence of the resputtering effect during the ion plating process. The FPSC with the ion-plated IWO electrode showed comparable performances to a commercial amorphous ITO electrode in an FPSC with an open circuit voltage (0.837 V), short circuit current (18.67 mA/cm$^2$), fill factor (72.54%), and power conversion efficiency (11.33%). Moreover, the microstructure and interfacial structure of the APIP-grown IWO film employed in a FPSC were examined by high-resolution transmission electron microscopy and the sheet resistance of the IWO films was correlated to the FPSC performance. © 2018 Author(s).

Perovskite solar cells with a methylammonium lead halide and mixed halide CH$_3$NH$_3$PbX$_3$ (MAPbX$_3$, X = Cl, Br, I) active layer have been considered as next-generation cost-efficient photovoltaics due to their attractive features including a high power conversion efficiency, light weight properties, simple device structure, and printing-based low cost fabrication.1–4 In addition, flexible or curved perovskite solar cells (FPSCs) can be simply realized due to the outstanding flexibility of all layers of the FPSCs except for transparent conducting oxide (TCO) electrodes and the low temperature coating process, which is suitable for the temperature limits of flexible substrates. However, the brittleness of ceramic TCO electrodes and the high-temperature coating process of the TCO electrodes are important hurdles of FPSCs. Although several flexible metal-, organic-, and carbon-based flexible electrodes have been employed in FPSCs, most FPSCs in both academic and industrial research still use an amorphous Sn-doped In$_2$O$_3$ (ITO) electrode prepared by DC sputtering as anode electrodes due to its low resistance, high transmittance, and easy scalability.5–14 Compared to an ITO electrode coated on a glass substrate, room temperature processed ITO films on a flexible substrate showed a fairly lower sheet resistance and higher optical transmittance owing to the insufficient Sn activation energy and amorphous structure of the ITO films. Because the critical performance of FPSCs such as current density-voltage curves and mechanical stability are mainly affected by the electrical, optical, interfacial, and mechanical properties of TCO electrodes, the development of high-quality TCO electrodes with a low sheet resistance, high optical transmittance, good flexibility, and smooth surface morphology to replace conventional amorphous ITO is very important. In addition,
considering the process temperature limits of flexible substrates and large area availability of FPSCs, the coating process for the TCO electrode should be carried out at a low temperature, below the transition temperature of the flexible substrate, and should be applicable to large area coating. For these reasons, a very thick F-doped SnO$_2$ electrode grown by high temperature chemical vapor deposition cannot be employed as a flexible TCO for FPSCs.\textsuperscript{15} Although several low temperature-processed printing or coating technologies to prepare flexible electrodes have been extensively reported, there are still drawbacks such as complicated process requirements, difficulties for large area coating, and roll-to-roll processes.\textsuperscript{16–18} As a promising alternative to the conventional sputtering process, the arc plasma ion plating (APIP) technique has attracted great attention due to its interesting merits such as low-temperature processing, high-quality TCO films, fast coating process, the absence of the resputtering effect, availability to large areas, and the roll-to-roll coating process.\textsuperscript{19} In the APIP process, the accelerated ions, which sublimate from the TCO tablet result in enhanced adhesion and densification of films as well as crystallization even at room temperature. Despite the attractive merits of the low-temperature APIP process, investigations of applying APIP-grown TCO films as a flexible and transparent anode for FPSCs are still lacking.

In this work, we suggest the feasibility of the APIP process to prepare high quality flexible TCO electrodes at room temperature for FPSCs. In particular, we employed a W-doped In$_2$O$_3$ (IWO) tablet in the APIP process to grow high mobility IWO films instead of typical ITO films as a flexible and transparent anode for FPSCs. The performance of FPSCs fabricated on APIP-grown high mobility IWO and sputtered amorphous ITO films were compared to show the potential of the APIP-grown IWO electrode. In addition, the microstructure and interfacial structure of the FPSCs with an APIP-grown IWO electrode were examined using high-resolution transmission electron microscopy (HRTEM). Furthermore, we correlated the sheet resistance of the APIP-grown IWO electrode with the performance of the FPSCs.

Flexible IWO films were grown on PET substrates by using an in-line type vertical 5-generation APIP system (SNTEK:NPS) without intentional substrate heating. Figure 1(a) schematically illustrates the APIP process employed to deposit 100 nm thick flexible IWO films on a 125 µm thick PET substrate using a high-density plasma gun with picture of plasma beam. Unlike a typical top-down type APIP system, the high-density plasma beam in the in-line APIP is irradiated from the side to the vertically located IWO tablet.\textsuperscript{19–21} The PET substrate-attached glass tray was automatically loaded into the process chamber with a base pressure of 1.0×10$^{-6}$ Torr. For sublimation of the IWO tablet (1 at% W-doped 99 at% In$_2$O$_3$), a high-density plasma beam was irradiated from a cathode gun to the IWO tablet at a constant Ar/O$_2$ flow rate of 300/90 sccm and working pressure of 3 mTorr. By applying a power of 3 kW to the plasma gun, we deposited 100 nm thick flexible IWO films on the PET substrates. During the APIP process, the PET substrate was located 600 mm from the IWO tablet. The electrical and optical properties of the APIP-grown IWO films were examined by Hall measurements and a UV/visible spectrometer. The surface morphologies of the APIP-grown IWO and sputtered ITO films were compared by field emission scanning electron microscopy (FESEM) and atomic force microscopy (AFM). The microstructure and interfacial structure of the FPSCs were investigated by HRTEM. To correlate the performance of the FPSCs and properties of transparent electrodes, FPSCs were fabricated on APIP-grown IWO anodes using PEDOT:PSS (hole transport layer), a CH$_3$NH$_3$PbI$_3$ active layer, PCBM (electron transport layer), and a BCP/Ag cathode layer. The photocurrent density-voltage (J-V) curves were obtained using a Keithley 2400 source measurement unit.

Table I compares the sheet resistance, resistivity, mobility, and carrier concentrations of the APIP-grown IWO (100 nm) and sputtered ITO (100 nm) films grown on PET substrates at room temperature. Even with the same room-temperature process, the resistivity and sheet resistance of the APIP-grown IWO film are lower than those of commercial sputtered ITO films due to its much higher mobility. In general, the sputtered ITO film at room temperature showed a fairly low carrier mobility due to the existence of severe scattering sources such as a disordered amorphous matrix and high carrier concentrations.\textsuperscript{22} However, the higher mobility of the APIP-grown IWO films could be attributed to the existence of W$^{6+}$ dopant in the crystalline In$_2$O$_3$ matrix-like transition metal dopants such as Ti$^{4+}$, Zr$^{4+}$, Gd$^{3+}$, and Mo$^{6+}$ with a high Lewis acid strength (LAS).\textsuperscript{23–27} The W$^{6+}$ or W$^{4+}$ ions sublimated by a high-density plasma beam could replace the In$^{3+}$ sites in the crystalline In$_2$O$_3$ matrix.
and produce excess electrons in the IWO films. In addition, the $\text{W}^{6+}$ dopant reduced the scattering of electrons and increased the carrier mobility because the W dopant can polarize electronic charge from the valence band more strongly towards itself. For these reasons, we employed high-mobility IWO as a room-temperature processed TCO electrode in the APIP process because a low-process temperature is insufficient for the activation of typical TCO films for a high carrier concentration. Figure 1(b) compares the optical transmittance of the APIP-grown IWO and sputtered ITO films on PET substrates, excluding the transmittance of the PET substrate. The APIP-grown IWO film showed

![Diagram](image)

**FIG. 1.** (a) Schematics of the in-line type APIP system used for deposition of 100 nm thick flexible IWO films on a PET substrate and picture of plasma beam on IWO tablet. (b) Optical transmittance and (c) surface FESEM images of APIP-grown IWO and sputtered ITO films on PET substrates at room temperature. The inset pictures in (b) show flexed IWO and ITO films with high transparency.

|                        | Sheet resistance (Ohm/square) | Resistivity (Ohm-cm) | Mobility (cm$^2$/V-s) | Carrier concentration (cm$^{-3}$) |
|------------------------|------------------------------|----------------------|-----------------------|----------------------------------|
| APIP-grown IWO/PET     | 37.14                        | $3.714 \times 10^{-4}$ | 61.7                  | $2.72 \times 10^{20}$            |
| Sputtered ITO/PET      | 51.17                        | $5.117 \times 10^{-4}$ | 22.5                  | $5.43 \times 10^{20}$            |
a high optical transmittance of 96.0% at a wavelength of 550 nm and an average transmittance of 88.2% between wavelengths of 800 to 2,500 nm, even when prepared at room temperature. However, the sputtered ITO film on a PET substrate showed an optical transmittance of 85.8% at a wavelength of 550 nm and an average transmittance of 79.9% between wavelengths of 800 and 2,500 nm. The pictures in the inset compare the color and transparency of the flexed IWO/PET and ITO/PET samples. The flexible IWO/PET sample clearly shows the Sungkyunkwan University’s logo due to the high optical transparency in the visible wavelength region and good flexibility. Due to the better crystallinity and lower carrier concentration of the APIP-grown IWO films compared to sputtered ITO films, they showed a higher optical transmittance in the visible and NIR wavelength regions. The high NIR transmittance of the IWO electrode is beneficial for tandem-structured FPSC with an active layer absorbing NIR wavelength light. The surface FESEM images in Figure 1(c) reveal the morphologies of the APIP-grown IWO and sputtered ITO electrodes on PET substrates. Unlike the sputtered ITO film, which has a rough surface morphology caused by the resputtering effect, the APIP-grown IWO film showed a very smooth and featureless surface without surface defects. The atomically smooth surface morphology of the APIP IWO film is attributed to the high ion energy accelerated to the PET substrate and the absence of the resputtering effect. 

Figure 2(a) shows an enlarged TEM image obtained from the middle region of the APIP-grown IWO films. As confirmed from the XRD (not shown here) plot, the APIP-grown IWO film showed a well-developed crystalline structure with a (222) preferred orientation, even in the film prepared at room temperature. The strong spots in the fast Fourier transformation (FFT) pattern in the inset also confirmed the well-developed bixbyite structure of the room temperature processed IWO film on the PET substrate. The well-developed crystalline structure of the APIP-grown IWO film could be explained by the different growth mechanism of APIP compared to typical sputtering. Even in the room temperature APIP process, the most sublimated gases are positively ionized by passing the high-density plasma beam region, as shown in Fig. 1(a). Then, the positively ionized gases are strongly repelled from the IWO tablet and accelerated to the self-biased PET substrate. The high energy of the accelerated ions results in an enhancement of adhesion and densification of films as well as crystallization, even at room temperature. Figure 2(b) shows the outer and inner bending radius test results for the APIP-grown IWO film and sputtered ITO film with decreasing bending radius. Up to an outer bending radius of 6 mm, both the IWO/PET and ITO/PET samples showed a constant resistance change, indicating a constant current flow and no crack formation. However, at an outer bending radius of 5 mm, both samples showed an abrupt increase of resistance change because the tensile stress led to crack formation and disconnection of the IWO and ITO films. In general, tensile stress applied on a film results in peeling off of the films and disconnects the current path. In
FIG. 3. (a) Schematic fabrication process and picture of a FPSC on the APIP-grown IWO electrode. (b) Photocurrent density-voltage (J-V) curves and (c) energy level diagram for FPSCs with the APIP-grown IWO and sputtered ITO electrodes. The inset in (b) shows the flexed FPSCs with the APIP-grown IWO electrode.

the inner bending test, both the IWO and ITO films showed a similar critical bending radius of 2 mm, which is a much smaller critical radius than outer bending. Because compressive stress leads to hill lock or physically overlapped cracks, both samples had a small critical bending radius. Considering the application of FPSCs, a critical bending radius of 5 mm is sufficient to fabricate curved or flexed PSCs in roofs or windows for buildings and automobiles.

To investigate the feasibility of APIP-grown IWO films as a flexible electrode in a FPSC replacing conventional sputtered ITO films, we fabricated FPSCs on APIP-grown IWO films with a sheet resistance of 37.14 Ohm/square and optical transmittance of 96.0%. Figure 3(a) illustrates the fabrication process and shows a picture of FPSCs with the APIP-grown IWO electrode. The APIP-grown IWO anode was simply patterned by a metal shadow mask attached on the PET substrate during the APIP process. The IWO anode was first treated with UV/ozone for 30 min. Then, PEDOT:PSS (Heraeus, AI 4083) as a hole transport layer was coated at 5,000 rpm for 40 sec onto the UV/ozone-treated IWO anode, followed by annealing at 110 °C for 10 min. For the perovskite layer as an active layer, the CH$_3$NH$_3$PbI$_3$ perovskite solution was spin-coated on the PEDOT:PSS layer by a consecutive two-step spin-coating process at 500 rpm for 5 sec and 6,000 rpm for 45 sec. During the second spin-coating step, the film was treated with toluene drop-casting, follow by annealing at 100 °C for 10 min in a N$_2$ glove box. Next, 20 mg/ml PCBM as an electron transport layer was coated onto the perovskite films at 1,000 rpm for 60 sec. Finally, the metal electrodes containing BCP (3 nm)/Ag (100 nm) as a cathode layer were deposited by a thermal evaporation process. The active area of the FPSC was patterned by cathode mask as 4.64 mm$^2$. Figure 3(b) shows the photocurrent density-voltage (J-V) curves for FPSCs fabricated on the APIP-grown IWO and sputtered ITO electrodes. Table II summarizes the performances of the FPSCs with different flexible and transparent electrodes. Compared to the FPSC with the sputtered ITO anode, the FPSC with the APIP-grown IWO electrode showed a higher power conversion efficiency (PCE) due to the higher short circuit current (J$_{sc}$) and open circuit voltage (V$_{oc}$). The higher J$_{sc}$ could be attributed to the higher optical transmittance of the APIP-grown IWO electrode than the sputtered ITO anode between wavelengths of 800 and 2,500 nm. Generally, the J$_{sc}$ of FPSCs is closely related to incoming light arriving at the MAPbI$_3$ active region, passing through the transparent anode. Therefore, the higher optical transmittance of the APIP-grown IWO electrode led to an increase of the J$_{sc}$ value for the FPSCs. However, the FF value of the FPSC with the IWO electrode is slightly lower than that of the FPSC with the sputtered ITO anode due to the small active area of 4.64 mm$^2$ even though IWO has a lower sheet resistance. Furthermore, the higher PCE of the FPSC with the APIP-grown IWO electrode is related to the slightly
higher $V_{oc}$ due to the higher work function of IWO (4.85 eV) compared to ITO (4.65 eV). Based on the work function measured by KPFM (Kelvin probe force microscopy), the energy diagram of the FPSCs with different electrodes is illustrated in Figure 3(c). Due to the presence of an identical hole transport layer (PEDOT) whose energy level is well-matched with the CH$_3$NH$_3$PbI$_3$ active layer, there is no significant difference of the $V_{oc}$ values of the FPSCs with the different IWO and ITO electrodes.

To investigate the microstructure and interface structure of the FPSC with the APIP-grown IWO anode, TEM examination was carried out. Figure 4(a) shows the full device structure obtained from a cross-sectional TEM image of the FPSC with the APIP-grown IWO anode. The FPSC showed a typical structure of a transparent anode (IWO)/hole transport layer (PEDOT)/active layer (CH$_3$NH$_3$PbI$_3$)/electron transport layer (PCBM)/metal cathode (BCP/Ag). In this p-i-n structure, light passes through the APIP-grown IWO anode and a hole carrier was extracted through the IWO anode. From the cross-sectional TEM image, well-defined interfaces were observed on the APIP-grown IWO anode, indicating no interfacial reactions. In particular, the interface between the IWO anode and PEDOT is atomically smooth and flat without any interfacial reaction due to the high density of the APIP-grown IWO film. Figure 4(b) shows the enlarged HRTEM images obtained from the A-D lettered region in the cross-sectional TEM image. In the case of the IWO anode, it shows a well-developed polycrystalline structure with a strong (222) preferred orientation on the PET substrate, as confirmed in Figure 2(a), even though it was grown at room temperature. The strong spots in the FFT pattern obtained from the bottom region of the IWO film indicate that the APIP-grown IWO film on the PET substrate has good crystallinity due to the high energy of sublimated ions during the APIP process. In particular, it is noteworthy that the interface between PEDOT and the IWO layer was very clean and there is no interfacial layer. The enlarged TEM image obtained from the CH$_3$NH$_3$PbI$_3$ active layer showed a single crystalline structure which is confirmed by the strong spots in the inset FFT pattern. The enlarged interface images between CH$_3$NH$_3$PbI$_3$ and the PCBM organic layer in Figure 4(b) show that an amorphous PCBM layer existed on the crystalline CH$_3$NH$_3$PbI$_3$ layer. As a result, the overall photovoltaic characteristics of the FPSC on the APIP-grown IWO electrode were better than those of the FPSC on the sputtered ITO electrode due to the good crystallinity and smooth morphology of the APIP-grown IWO electrode caused by high energy ion acceleration into grown IWO films during the room temperature APIP process. This indicates that the APIP-grown high mobility IWO electrode is a promising replacement for sputtered amorphous ITO electrodes due to its low resistance, high optical transmittance, and good mechanical properties as well as compatibility for commercial large area coating or roll-to-roll coating such as typical sputtering.

Better performance of the FPSCs with the APIP-grown IWO films also can be expected based on the series resistance of transparent conducting electrodes. Figure 5 shows the three-dimensional and cross-sectional structures of the FPSCs fabricated with the APIP-grown IWO electrode to show hole
FIG. 5. (a) Three-dimensional and (b) cross-sectional structures of the FPSCs with the APIP-grown IWO films to calculate the series resistance component for the transparent conducting electrode.

The generated currents flow perpendicular to the CH$_3$NH$_3$PbI$_3$ active layer which could be extracted through the APIP-grown IWO electrodes, as indicated by the arrows. The series resistance component for the APIP-grown IWO electrodes could be calculated using some assumptions. First, the currents flow uniformly over the active area of the FPSCs. Second, the lateral currents in the APIP-grown IWO electrodes that are a distance (x) away from the collecting contact were obtained by integrating the current density (J) from the distance (x) to the edge of the FPSCs (β) as follows:

$$I_{IWO}(x) = \int_x^\beta Jdx = J(\beta - x), \quad (1)$$

Where J is the current density from the active layer of the FPSCs and the β is the length of the BCP/Ag electrodes of the FPSCs. The voltage drops ($V_{IWO}$) introduced by the currents collected through the APIP-grown IWO layer can be calculated from the product of the current distribution ($I_{IWO}$) and the incremental resistance ($dV=I\,dR$) as follows:

$$V_{IWO} = \int_0^\alpha I_{IWO}(x)\,dR = \int_0^\alpha I_{IWO}(x)\frac{\rho_{IWO}}{t}\,dx = \int_0^\alpha J(\beta - x)\frac{\rho_{IWO}}{t}\,dx = J\frac{\rho_{IWO}}{t}\left(\beta - \frac{\alpha}{2}\right), \quad (2)$$

where $\rho_{IWO}$ is the electrical resistivity of the APIP-grown IWO electrodes and t is the thickness of the APIP grown IWO electrodes. Also, β is larger than $\alpha/2$ ($\beta > \alpha/2$). Finally, the series resistance component of IWO can be expressed by the following equation:

$$R_{IWO} = \frac{V_{IWO}}{J} = \frac{\rho_{IWO}}{t}\left(\beta - \frac{\alpha}{2}\right). \quad (3)$$

As shown in equation (3), the series resistance of the FPSCs is directly proportional to the electrical resistivity of the electrodes. Therefore, better performance of the FPSCs with the APIP-grown IWO film could be attributed to the lower resistivity of the APIP-grown IWO film compared to that of the sputtered ITO films.

In summary, we evaluated the electrical, optical, and mechanical properties of APIP-grown IWO electrode on PET substrates at room temperature as a replacement of conventional amorphous ITO/PET for high performance FPSCs. Due to easy crystallization and the W dopant screening effect, the APIP-grown IWO showed a low resistivity of 3.714×10$^{-4}$ ohm-cm and high optical transmittance of 96.0% for fabrication of FPSCs. A different growth mechanism of the APIP process than that of the typical sputtering process led to the formation of a well-developed crystalline IWO film on the PET substrate even at room temperature. The higher power conversion efficiency of the FPSC with the APIP-grown IWO electrode than the ITO-based FPSC indicates that APIP-grown IWO is a promising room-temperature processed electrode material and coating technology for high performance FPSCs.

This work was supported by a National Research Foundation of Korea (NRF) grant funded by the Korean government (MSIP) (No. 2018R1A2B2003826) and partially supported by Korea Electric Power Corporation (KEPCO, CX72170049).
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