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Synthesis and characterization of electro-spun TiO$_2$ and TiO$_2$-SnO$_2$ composite nano-fibers for application in advance generation solar cells

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Abstract

Due to rapid progressive research in the field of nano-technology, nanostructures are attaining tremendous deliberation. Precisely, among these, nano-fibers have accumulated unprecedented attention over the last few years owing to their superior properties such as large surface area to volume ratio. For advance generation solar cells, nano-fibers of metal oxides such as TiO$_2$ have been widely employed as a working anode but due to the disadvantages of recombination of electrons, their coupling with other metal oxides is necessary to render them useful. This work reports the successful fabrication of TiO$_2$-SnO$_2$ nano-fibers based film and study of the effect of polymer concentration upon it. The crystal structure of synthesized fibers was investigated using XRD, the morphology and chemical composition was analyzed using SEM and EDX, optical properties and chemical properties were studied using UV-VIS spectrophotometer and FT-IR while the electrical properties were studied using Hall Effect measurement system. It was revealed that the prepared TiO$_2$-SnO$_2$ nano-fibers exhibit enhanced conductivity, larger band-gap with enhanced photo voltaic properties than TiO$_2$ nano-fibers. The prepared TiO$_2$/SnO$_2$ nano-fibers based solar cells provide an improved efficiency of 4.81% as compared to 1.8% efficiency of bare TiO$_2$ nano-fibers.

1. Introduction

Material limitations of wafer-based silicon (Si) cell technology and efficiency limitations of thin film solar cell technologies need to be overcome to achieve affordable PV technology [1]. To prevail over these hindrances, advance generation solar cells are a winsome choice. This generation of solar cell encompasses various advantages that can be called upon to expand the range of applications where conventional solar cells are unsuitable. These applications comprises of low light performance, versatile product integration, low energy manufacturing cost [2].

Initially advance generation solar cells utilize bulk semi-conductor materials as their working anode. Bulk semi-conductor materials show photo-corrosion when exposed to sun-light, which decrease the stability of the cells. To reduce this issue, large band-gap nano-structured semi-conductors are employed. These nano-structured semi-conductors exhibit resistance towards photo-corrosion, spectacular optical and electrical properties. Among these nano-structures, nano-fibers have attained marvelous attention in past few years owing to their superior properties. Nano-fibers have found various applications in a variety of fields ranging from medical to high-technology. In advance generation solar cells, metal oxide based nano-fibers are widely used because of their large surface area, high electrical conductivity, fast and unhindered electron transport from photon absorber to FTO, better sensitizer loading and hence improved light-harvesting properties [3].
Metal oxides including TiO$_2$, SnO$_2$, ZnO and Nb$_2$O$_3$ has been widely grown into such one dimensional nano-structures. Among these oxides, TiO$_2$ has been extensively employed as electron transport material in most of the advanced generation solar cells mainly Dye-sensitized solar cells and other hybrid and oxide solar cells. According to Kim et al., nano-fibers of TiO$_2$ possess significant superiority over other nano-structures in term of providing longer one dimensional path for charge transfer, but the increased possibility of recombination of charge carriers results in the decline in efficiency. Coupling of TiO$_2$ nano-fibers with other suitable materials can improve its photo-catalytic activities because of the enhancement of charge separation. Various semiconductor to semiconductor based hetero-structures of TiO$_2$ are currently being used including TiO$_2$/SnO$_2$, ZnO/SnO$_2$ and TiO$_2$/ZnO. Among these hetero-structures, TiO$_2$/SnO$_2$ manifests several advantages over others because of the low cost of SnO$_2$ and some precise crystallographic planes of nano-crystals of TiO$_2$ and SnO$_2$. The presence of Sn in the synthesis invariably lowers the average crystallite size. This effect is mainly due to the prevailing surface location of the metal species, which can inhibit the crystal growth. When UV light falls on such structures with photon of energy higher than or equal to the band gaps of TiO$_2$ and SnO$_2$, the photo-generated electrons in the irradiated TiO$_2$ can move to the conduction band (CB) of SnO$_2$ and the photo-generated holes in the irradiated SnO$_2$ move to the valence band (VB) of TiO$_2$ as shown in figure 1. As a result, TiO$_2$/SnO$_2$ hetero-structures could hinder the charge recombination and improve the device efficiency. Moreover, band-gap can also be adjusted by using hetero-structures.

Nano-fibers can be sensitized using various methods including template-assisted synthesis, chemical vapor deposition (CVD), self-assembly, wet chemical synthesis, and electro-spinning. Electro-spinning is the broadly used technique for nano-fibers synthesis which is further divided into various methods including melt-electro-spinning, co-axial electro-spinning, magnetoelectro-spinning depending upon the solution and required morphology. Among these synthesis techniques, co-axial electro-spinning utilizes two different precursor solutions that come into contact at the tip and are ejected out of the same needle with high voltage which stretches the droplet into a taylor cone resulting in the formation of core-shell nano-fibers. Electro-spinning utilizes polymer as binding material for proper stretching of nano-fibers. Concentration of the polymer solution critically affects the nano-fibers structure. The aim of this study is to Fabricate, characterize TiO$_2$ nano-fibers and SnO$_2$/TiO$_2$ nano-fibers using co-axial electro-spinning and studied its application in dyesynthesized solar cells. Analysis of morphological, structural, optical as well as electrical properties of the prepared fibers was performed to evaluate their electron transport ability. Study of effect of polymer concentration on structure of prepared fibers was also carried out in this work.

2. Experimental

2.1. Materials
Titanium iso-propoxide (C$_{12}$H$_{28}$O$_4$Ti), Poly-vinyl pyrrolidone (C$_6$H$_9$NO))$_n$ (M.W = 3,000,000 g.mol$^{-1}$), Stannous chloride di-hydrate SnCl$_2$.2H$_2$O, Absolute Ethanol (C$_2$H$_5$OH), glacial acetic acid (CH$_3$COOH) were used as raw materials in this study.
2.2. Synthesis of bare TiO2 nano-fibers and TiO2/SnO2 nano-fibers
Titanium iso-propoxide was used as a precursor for synthesizing TiO2 nano-fibers. Stannic chloride di-hydrate was used as SnO2 nano-fibers precursor. At first, a homogeneous solution of Ethanol and Glacial acetic acid was made in a ratio of 4:1, by magnetically stirring for 10 min. Various PVP concentrations were added as shown in Table 1 to the solution to get a viscous polymeric solution. After stirring the polymer solution for 1 h, 0.5 g of titanium iso-propoxide was added to the solution resulting in a colour transformation to light yellowish hue. The solution was stirred until a homogenous mixture was formed. Following the same methodology such as in the case of synthesis of TiO2 nano-fibers, SnO2 nano-fibers solution was synthesized by adding 0.2 g of Stannic chloride di-hydrate instead of Titanium iso-propoxide. The solution was stirred until a homogenous mixture was obtained. Different solutions with variable polymer concentration were prepared, as reported in Table 1.

### Table 1. Prepared Solutions which variable polymer concentrations.

| Sample | Ethanol: Glacial Acetic Acid | Titanium iso-propoxide | Stannic chloride Di-hydrate | PVP |
|--------|-----------------------------|------------------------|----------------------------|-----|
| T1     | 1:1                         | 0.5 ml                 | —                          | 1.4 g |
| T2     | 1:1                         | 0.5 ml                 | —                          | 1.6 g |
| T3     | 1:1                         | 0.5 ml                 | —                          | 1.8 g |
| TS1    | 1:1                         | 0.5 ml                 | 0.4 g                      | 1.4 g |
| TS2    | 1:1                         | 0.5 ml                 | 0.4 g                      | 1.6 g |
| TS3    | 1:1                         | 0.5 ml                 | 0.4 g                      | 1.8 g |

2.2.1. Electro-spinning setup
The conductive glass (FTO) of dimension of 2 cm² x 2 cm² was first cleaned thoroughly by sonication in distilled water followed by sonication in ethanol and acetone for 10 min. Cleaned FTO is attached on the top of Aluminium foil which is placed in front of the needle on the collector plate via a conductive carbon tape.

2.2.2. Electro-spinning of solutions
For core-shell nano-fibers, 2 solutions are made which are then subjected to different syringes for nano-fiber synthesis. The highly viscous polymeric solution of TiO2 and SnO2 were inserted into 5 ml syringes in a horizontally attached electro-spinning setup. A high voltage of 17.8 kV was attached to this assembly and the solution was injected to the needle at the feed rate of 750 μl h⁻¹. The needle collector distance was set and fixed at 15 cm. A stable taylor cone was acquired which was clearly visible at the tip of the needle. The sample was deposited for 45 min at ambient temperature. The as-spun fibers deposited on FTO and aluminium foil were dried for 1 h at 100 °C in a vacuum oven. The heating rate was set at 1 °C/min. The dried nano-fibers were then calcined at 500 °C for 1 h in a tube furnace in air. To attain the calcination temperature a heating rate of 3 °C/min was maintained inside the placement chamber. For both Bare TiO2 nano-fibers and TiO2/SnO2 core shell nano-fibers, 17.8 kV of voltage was applied. The schematic figure of the fabrication of TiO2/SnO2 core shell nano-fibers is shown in figure 2. For device fabricated, the prepared nano-fibers of TiO2 and TiO2-SnO2 composite nano-fibers were coated on indium tin Oxide (ITO) glass having a thin spin coated layer of TiO2 nano-particles. This thin layer provides better electron transport. The prepared anode was furthermore dipped into N3 dye solution for 20 h for better dye absorption. An ITO glass with a thin layer of gold is used as a counter electrode. The photo-anode with dye is clipped with the gold counter electrode. In the space between the electrodes, a redox electrolyte (I-/I3) solution was inserted using capillary action. Three set of cells were fabricated for TiO2 and TiO2-SnO2 composite nano-fibers to check repeatability.

2.2.3. Characterization
The electrical conductivity, optical properties, structure and morphology of TiO2 nano-fibers were characterized by different techniques. The morphology was imaged by using scanning electron microscope (SEM) (Model: Vega3, Tuscan) and elemental composition of the samples was analysed using energy dispersive x-ray spectroscopy. The crystallinity of nano-fibers under consideration was studied using x-ray diffractrometer (Model:Advanced D8,Bruker). The electrical conductivity of the prepared film was measured using Hall Effect measurement system (Ecopia, HMS-3000) while UV-3600 plus UV-VIS-NIR Spectrophotometer was used to study its optical properties. For IV measurements, potentiostat system (Biological VSP) was used with a working distance of 8 cm for light intensity similar to sun light.
3. Results and discussions

3.1. Morphological and Elemental Analysis

The high resolution SEM micrographs of TiO$_2$ and TiO$_2$/SnO$_2$ nano-fibers at various polymer concentrations are shown in figures 2 and 3. The average diameter of the finally prepared TiO$_2$ and TiO$_2$/SnO$_2$ nano-fibers is less than the electro-spun nano-fibers before annealing. This effect is perceptible due to the evaporation of solvent and PVP after annealing. During the calcination process, all the organic contents in electro-spun fibers decomposes [26] resulting in morphological change from fibrous to hollow structure [27]. The diameter of TiO$_2$ nano-fibers and core-shell TiO$_2$/SnO$_2$ nano-fibers with 1.4 g, 1.6 g and 1.8 g ranges from 38-48 nm, 50-63 nm, 60-85 nm and 60-120 nm, 70-145 nm and 80-171 nm respectively as shown in figure 5.

As seen in figures 2(a) and 3(a) fibers diameter is smaller however there are various bead structures which are produced due to the high surface tension of the polymer solution. With increase in polymer concentration the Electro-spun solution cause rise in the viscosity of the solution which could persuade the formation of fibers with wider diameter [28]. At high viscosity of the solution, formation of jet is hard which results in the formation of inadequate stretching of the electro-spin solution. This results in the production of wider diameter fibers. Less diameter exhibiting fibers are assumed to offer large surface area to volume ratio, which is expected to reveal better dye absorption in solar cells [29]. Since the dye monolayer is adsorbed on the semiconductor oxide; it is noticeable that its high surface area will enhance the light absorption. The high surface area of nano-structures, there would be an increase in the light scattering [30].

The round-like/spherical structure confirms the formation of core-shell nano-fibers as shown in figures 4(a)–(c). The un-annealed nano-fibers of TiO$_2$/SnO$_2$ nano-fibers in sample with PVP concentration of 1.8 g is shown in figure 5. The fibers are smooth and uniform. There average diameter in this case is larger than the annealed sample. This effect is mainly due to the presence of polymer and other solvent based materials in
the fibers. After annealing process, only the required metal-oxide based nano-fibers are left. Figure 6(a), (b) shows a plot between the diameter of the fibers with change in polymer concentration.

The presence of Sn in TiO2/SnO2 nano-fibers can also be seen in EDS and FT-IR results. The EDS pattern shows prominent peaks of Ti, Sn and O in case of TiO2/SnO2 nano-fibers as shown in figure 7 without traces of any impurities. No traces of carbon in the spectrum show that the PVP is removed completely after annealing process. Presence of high percentage of Sn as shown in table 2, depicts that SnO2 is successfully encapsulated in TiO2 nano-fibers and of core-shell structure of SnO2 and TiO2 are created [31].

The predictable formation of nano-fibers depends on the experimental mechanism. Among the solvents, glacial acetic acid is used as a catalyst and PVP polymer is used as a binder materials. Polymer materials are however used to give the required molecular weight and concentration for proper stretching of the solution to be electro-spun. Following reaction occurs during the formation of TiO2 nano-fibers.

\[
\text{Ti(OH)}_4 + 4\text{R'OH} \rightarrow \text{Ti(OH)}_4 + 4\text{ROH'}
\]

Following reaction occurs during the formation of SnO2 nano-fibers.

\[
\text{SnCl}_2\cdot 2\text{H}_2\text{O} + \text{ROH} \rightarrow \text{Sn(OH)}_4 + \text{RCl}
\]

\[
\text{Sn(OH)}_4 \rightarrow \text{SnO}_2 + \text{H}_2\text{O}
\]

X-ray Diffraction (XRD) of the synthesized nano-fibers was carried out in order to investigate the crystal phase of TiO2 and TiO2/SnO2 nano-fibers. Figure 8(a) shows the XRD pattern of bare TiO2 nano-fibers after drying at 100 °C. No evident peaks can be seen in the spectrum, which shows the amorphous behaviour of the TiO2 nano-fibers film. Similarly the polymer (PVP) has not been evaporated properly. However, figure 8(b)
demonstrating evident major anatase peaks of TiO$_2$ as per JCPDS card number 21-1272. The peak at $2\theta = 25.8^\circ$ confirms the formation of TiO$_2$ anatase structure [32]. The XRD pattern of bare TiO$_2$ nano-fibers depicts distinctive peaks at $2\theta = 25.8^\circ$, 37.7$^\circ$, 48.01$^\circ$, 53.8$^\circ$, 55.03$^\circ$ and 62.10$^\circ$ which can be assigned to (101), (112), (200), (105), (211) & (204) planes respectively with tetragonal anatase structure. Anatase phase of TiO$_2$ inherently demonstrate properties like large mobility and wider optical absorption gap. Figure 8(c) shows the addition of 40% SnO$_2$ in anatase phase of TiO$_2$. The prominent peaks of (110), (101), (211) and other small peaks in figure 7(c) match with the corresponding peaks of tetragonal rutile phase of SnO$_2$ (JCPDS 41-1445). No additional impurity phase was found, validating the existence of material as a pure nano-composite [33].

Figure 9(a) demonstrated the FTIR spectra of TiO$_2$/SnO$_2$ nano-fibers before and after calcination. There are several sharp peaks before annealing. These peaks can be attributed to the presence of polymer and solvent present before annealing. A prominent peak is seen at 1655 cm$^{-1}$ which represent the functional unit of C=O in PVP [33]. The other peak at 1277 cm$^{-1}$ represent C-N bond stretching mode [34]. Moreover peak at around 1400 cm$^{-1}$ represent vibration of CH$_2$ bond of PVP. A small peak at around 1050 cm$^{-1}$ represents the vibration

| Atom % | Ti   | Sn  | O   |
|--------|------|-----|-----|
| TiO$_2$ Nano-fibers | 12.15 | —   | 87.85 |
| TiO$_2$/SnO$_2$ Nano-fibers | 9.22  | 7.86 | 82.92 |

Figure 6. Fiber diameters with various PVP concentrations (a) TiO$_2$ nano-fibers (b) TiO$_2$—SnO$_2$ composite nano-fibers.

Figure 7. EDX Spectra of annealed (a) TiO$_2$ nano-fibers (b) TiO$_2$/SnO$_2$ nano-fibers.
bend of Ti–O–C. A broad peak in between 3250 cm⁻¹ and 3600 cm⁻¹ shows the absorption of moisture and solvent during the process of electro-spinning. After annealing there is no major peak in FTIR spectra implying that no impurity exists after the heat treatment [35]. This validate that no water, hydroxyl and hydrocarbon impurity is detected in the IR spectra of calcined TiO₂/SnO₂ nano-fibers [36].

3.2. Optical and electrical properties analysis

The optical properties were studied in wavelength range of 300 nm to 800 nm using UV-VIS NIR spectrophotometer. In the absorption spectrum of TiO₂-SnO₂ nano-fibers, a blue shift was observed as compared to TiO₂ nano-fibers; such a shift can be attributed to the incorporation of SnO₂ [36].

The band-gap (E_g) of the prepared materials was calculated by using tauc plot; αhv = A(hv-E_g)ⁿ [37], where A is the absorption coefficient calculated from UV VIS absorption spectra shown in figure 10, h is the plank constant, ν is the frequency which is equals to ν = 3 × 10⁸/λ, A is a constant and n = 1/2 referred to direct allowed transition. Tauc plot is drawn between the photon energy hv (eV) and direct band-gap (αhv)². The calculated band-gap of TiO₂-SnO₂ nano-fibers is 3.81 eV as compared to the band-gap of TiO₂ nano-fibers of 3.52 eV. This increase in band-gap means increase in Fermi level and open circuit voltage for a typical DSSC solar cell. Moreover it facilitates electron transfer from excited state of electron to the external circuit [38].
3.3. Electrical measurements

The Electrical property including conductivity of the annealed nano-fibers deposited on FTO (conductive glass) was studied using Hall Effect measurement system. The conductivity of TiO$_2$-SnO$_2$ nano-fibers is found to be higher than that of bare TiO$_2$ nano-fibers as shown in Figure 11. The conductivity of TiO$_2$ nano-fibers and TiO$_2$/SnO$_2$ nano-fibers was calculated to be $1.831 \times 10^{-2}$ S.m and $1.407 \times 10^{-1}$ S.m respectively. This is primarily because of the addition of SnO$_2$ to TiO$_2$ nano-fibers. Addition of SnO$_2$ stabilizes the anatase phase of TiO$_2$.

Similarly with addition of SnO$_2$ the number density of the fibers increases, as a result of rise of the fibers with variable diameter. The hetro-structure of TiO$_2$/SnO$_2$ will also facilitate charge separation which results in the reduction of recombination of electrons. The electrons in TiO$_2$ will flow to SnO$_2$, because the work function of TiO$_2$ is smaller than SnO$_2$. This phenomenon can result in an increase of the electrons concentration in the SnO$_2$ layer, which results in an increase in conductivity of the film. Similarly with increase in nano-fiber diameter, the conductivity of the film decrease as shown in Figure 10. The decrease is conductivity can be attributed to the geometric surface and packing density effect. The working anode of solar cell should have high conductivity to facilitate the movement of electron from photon absorber to the external circuit easily [39].

3.4. Photovoltaic performance of DSSC

The effect of nano-fibers diameter on photovoltaic performance of DSSC was studied. The fill factor and the efficiency of the prepared cell were calculated using the $V_{oc}$ and $J_{sc}$. As shown in figure 12, thinner diameter nano-fibers based DSSC shows better performance due to larger surface area to volume ratio. The results in better dye absorption and more efficiency of electron transfer. The large beads form in high viscous solution results in light scattering which produce less efficient solar cells. Moreover, with the incorporation of SnO$_2$ in TiO$_2$ nano-fibers, efficiency increases. This effect is due to the better charge separation for electron transport throughout the circuit.
By calculating Fill factor the efficiency of the in house prepared solar cells was studied. The table 3 shows that a change in the fiber diameter results in a change in efficiency of the cell. For sample T1 with 1.2 g PVP concentration, the overall efficiency of the cell is 1.8%. However with increase in polymer concentration to 1.8 g, the efficiency decreases to 1.33%. This effect is because of the beads produced due to high polymer concentration. However by the addition of SnO2 to TiO2 nano-fibers, the overall solar cell efficiency can be enhanced to 4.81%. However, the effect of polymer concentration is also valid in TiO2/SnO2 core shell nano-fibers. The increase in the value of Jsc depicts a better diffusion and injection of electrons which provide better dye absorption and electron transfer in advance generation solar cells.

Table 3. Comparison of photo-voltaic parameters (Voc , Jsc, FF and η) for TiO2 and TiO2/SnO2 nano-fibers samples with different PVP concentration.

| Sample | Voc (V) | Jsc (mAcm⁻²) | Fill factor | Efficiency η(%) |
|--------|---------|---------------|-------------|-----------------|
| T1     | 0.75    | 6             | 0.4         | 1.8             |
| T2     | 0.74    | 5.9           | 0.38        | 1.63            |
| T3     | 0.72    | 5.9           | 0.29        | 1.33            |
| TS1    | 0.81    | 8.4           | 0.70        | 4.81            |
| TS2    | 0.8     | 8.2           | 0.70        | 4.6             |
| TS3    | 0.78    | 8             | 0.69        | 4.46            |

Table 4. Benchmark table between Literature and this work.

| Reference   | Material                    | Jsc (mAcm⁻²) | Voc (V) | Efficiency η(%) |
|-------------|-----------------------------|--------------|---------|-----------------|
| PDu (2014)  | TiO2 Nano-particles/Nano-fibers | 10.6         | 0.70    | 4.46            |
| Wali (2016) | SnO2 nano-fibers/TiO2 nano-fibers | 18.0         | 0.57    | 4.5             |
| Lim (2016)  | Hierarchical TiO2–SnO2 Nano-composite | 12.68        | 0.61    | 3.73            |
| Zhang (2016)| Mesoporous TiO2 nano-fibers  | 3.937        | 0.915   | 1.82            |
| Altaf (2019)| TiO2 nano-fiber             | 8.99         | 0.738   | 4.09            |
| This work   | TiO2/SnO2 composite core shell nano-fibers | 8.4          | 0.81    | 4.81            |

By calculating Fill factor the efficiency of the in house prepared solar cells was studied. The table 3 shows that a change in the fiber diameter results in a change in efficiency of the cell. For sample T1 with 1.2 g PVP concentration, the overall efficiency of the cell is 1.8%. However with increase in polymer concentration to 1.8 g, the efficiency decreases to 1.33%. This effect is because of the beads produced due to high polymer concentration. However by the addition of SnO2 to TiO2 nano-fibers, the overall solar cell efficiency can be enhanced to 4.81%. However, the effect of polymer concentration is also valid in TiO2/SnO2 nano-fibers. The increase in the value of Jsc depicts a better diffusion and injection of electrons which provide better dye absorption and electron transfer in advance generation solar cells.

Table 4 shows a comparison between this work and the latest literature. The core shell nano-fibers based DSSC in comparison with other literature shows an enhanced power conversion efficiency of 4.81%. The efficiency can furthermore be enhanced by studying the effect of change of SnO2 precursor concentration in TiO2/SnO2 core shell nano-fibers based DSSC. Moreover the effect of change of diameter mainly due to other process parameters including voltage, flow rate, collector to needle distance can also be studied.
4. Conclusion

In this work, TiO$_2$ and TiO$_2$-SnO$_2$ nano-fibers were successfully synthesized using solution electro-spinnning and co-axial electro-spinnling methods, to be employed as active anode for advance generation solar cells. The diameter of the fibers is found to be less than 200 nm. XRD Pattern confirms the formation of anatase TiO$_2$ and tetragonal rutile phase of SnO$_2$. Furthermore, the morphological analysis reveals the hollow structure of nano-fibers. Study of effect of polymer concentration upon nano-fibers synthesis demonstrated that increase in polymer concentration results in the increase in diameter by approximately a quarter. FTIR results validated that no water, hydroxyl group or hydrocarbons are present after annealing. Moreover, UV-VIS spectral data signifies that there has been a significant increase in band-gap in case of TiO$_2$-SnO$_2$ nano-fibers in comparison with bare TiO$_2$ nano-fibers. The electrical properties of the prepared films calculated using Hall Effect measurement also showed promising results denoting high conductivity values of TiO$_2$-SnO$_2$ nano-fibers film with an efficiency of 4.81% which making them undoubtedly suitable for future solar cell technologies and applications.

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