Efficient Performance of Electrostatic Spray-Deposited TiO$_2$ Blocking Layers in Dye-Sensitized Solar Cells after Swift Heavy Ion Beam Irradiation

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Abstract

A compact TiO$_2$ layer (~1.1 μm) prepared by electrostatic spray deposition (ESD) and swift heavy ion beam (SHI) irradiation using oxygen ions onto a fluorinated tin oxide (FTO) conducting substrate showed enhancement of photovoltaic performance in dye-sensitized solar cells (DSSCs). The short circuit current density ($J_{sc} = 12.2$ mA cm$^{-2}$) of DSSCs was found to increase significantly when an ESD technique was applied for fabrication of the TiO$_2$ blocking layer, compared to a conventional spin-coated layer ($J_{sc} = 8.9$ mA cm$^{-2}$). When SHI irradiation of oxygen ions of fluence $1 \times 10^{13}$ ions/cm$^2$ was carried out on the ESD TiO$_2$, it was found that the energy conversion efficiency improved mainly due to the increase in open circuit voltage of DSSCs. This increased energy conversion efficiency seems to be associated with improved electronic energy transfer by increasing the densification of the blocking layer and improving the adhesion between the blocking layer and the FTO substrate. An increase in the electron transport from the blocking layer may also retard the electron recombination process due to the oxidized species present in the electrolyte. These findings from novel treatments using ESD and SHI irradiation techniques may provide a new tool to improve the photovoltaic performance of DSSCs.

Introduction

Dye-sensitized solar cells (DSSCs) are a promising photovoltaic system for next generation solar cells that contain mesoporous nanocrystalline semiconductors like TiO$_2$, ZnO and SnO$_2$ as photoanodes anchored with dye molecules. These dye molecules serve as light harvesters [1-3]. It is believed that DSSCs are more cost effective than conventional solar cells due to their low production cost. Recently, intensive research activities have focused on enhancing the photoconversion efficiency of DSSCs by improving charge transport in the electronic interfaces such as (a) TiO$_2$/transparent conducting oxide (b) TiO$_2$/electrolyte (c) dye/TiO$_2$ (d) dye/electrolyte and (e) electrolyte/counter electrode. For instance, electrons on either side of the TiO$_2$ layer or in the transparent conducting oxide (TCO) such as fluorinated tin oxide (FTO) may recombine with the oxidized redox couples such as I$_3^-$.

Electron recombination is one of the major factors that determine the high energy conversion efficiency ($2e^- + I_3^- \rightarrow 3I^-$) [4,5]. Therefore, there have been several different approaches to reduce or block the recombination of electrons on TCO and TiO$_2$ layers to improve the energy conversion efficiency. Among the interfaces described previously, the one between TiO$_2$/transparent conducting oxides faces severe recombination problems, since the porous nature of photoanodes results in uncovered sites on the TCO layer, resulting in sites for electron recombination with I$_3^-$ redox species in the electrolyte.

Considerable attention has been focused on the methods to reduce electron recombination at the interface between TCO substrate and electrolyte containing I$_3^-$. In order to overcome this recombination problem, a compact oxide layer (pore-free and dense) is commonly introduced between the mesoporous TiO$_2$ and the TCO substrate, which blocks electron recombination with the electrolyte via a so-called blocking effect [6]. Furthermore, the blocking layer should provide good adhesive properties between the TCO and the mesoporous TiO$_2$.
layers to facilitate electron transport from the mesoporous TiO₂ to the TCO layers. From this perspective, a variety of oxides have been investigated such as Nb₂O₅ [7], ZnO [8], MgO [9], Al₂O₃ [10] and SiO₂ [11] in addition to TiO₂ [12]. Different preparation techniques have been widely exploited to form blocking layers such as sol-gel [12], spin coating [13], sputtering [14,15] and spray-coating [16] techniques. Therefore, the formation of a blocking layer between mesoporous TiO₂ and the TCO substrate has been investigated, which not only blocks electron recombination but also facilitates electron transport.

In this study, electrostatic spray deposition (ESD) was applied first for fabricating a TiO₂ blocking layer, and swift heavy ion beam irradiation (SHI) was subsequently performed as a post-treatment, since ESD allows particle size and shape to be controlled by varying processing parameters such as the polymer concentration in the spray solution and applied voltage. Furthermore, a conventional electrospinning setup, in which the conducting FTO electrode directly connected to the electric circuit (negative terminal) may produce an electrohydrodynamic field between a collector (FTO) and a sol injector (syringe), may improve adhesion between the sprayed particles and the FTO substrate. Particle growth achieved via ESD is more effective than that obtained by conventional spray pyrolysis [17] or spin coating. Chen et al. [18] reported nanostructured TiO₂ films fabricated by ESD and studied their phase transformations by sintering. Zhang et al. [19] demonstrated the feasibility of ESD-derived uniform TiO₂ particles in DSSCs and suggested that the electrical contact between the conducting substrate and TiO₂ particle (electron transport layer) plays a crucial role in power conversion efficiency, since the presence and the removal of the polymer molecules in the ESD layer during sintering may result in poor contact among TiO₂ nanoparticles and poor adhesion to conductive glass substrates. These will impose severe constraints on the electron transport from the mesoporous TiO₂ layer to the FTO substrate. Therefore, an alternative post-treatment may be necessary to obtain a compact, thin blocking layer with good contact among TiO₂ nanoparticles and good adhesion to the conductive glass substrates [20], resulting in rapid electron transport. SHI was employed as a post-treatment for improving both adhesion and contact. Recently, Singh et al. [21] reported that SHI irradiation improved the transmittance of conducting substrates (indium-doped tin oxide), and their performance was affected in DSSCs. The SHI method is based on the interactions of ions with solids, where the temperature around the trajectory of the ion increases remarkably. The shock waves, or so-called pressure waves, develop due to the temperature spike, which diffuses the heat radially in the target [22]. This thermal spike can generate local heat along TiO₂ nanoparticles. When the temperature is greater than the melting temperature of TiO₂ (~1,300 °C), a liquid phase is formed in this specific region. This high temperature region cools down immediately due to very rapid heat transfer to the surroundings, resulting in solidification of the surface, specifically melted TiO₂ nanoparticles [23] that form a highly adhesive TiO₂ blocking layer with the FTO substrate. To best of our knowledge, this is the first report of its kind to apply the SHI irradiation technique for obtaining an efficient blocking layer in DSSCs. The performance of the SHI-irradiated blocking layer was investigated in comparison with the unirradiated (pristine) and conventional spin-coated TiO₂ blocking layers.

**Experimental**

The following procedure was used for the preparation of a TiO₂ blocking layer on fluorinated tin oxide (FTO) substrates: 15 wt% poly(vinyl acetate) (PVAc) (Mn ~ 5,000,000) solution was prepared by dissolving PVAc in dimethyl formamide (DMF) and dropping it into a mixture containing 1 g of titanium isopropoxide and 0.5 g of acetic acid while stirring. The as-prepared TiO₂ sol was electrospayed onto a grounded FTO substrate at 17 kV with a constant distance of about 10 cm between FTO and the electrospray syringe at a flow rate of 1.0 ml/h. The resultant ESD TiO₂ blocking layer was ~1.1 μm thick and was sintered at 450°C for 30 min in air. In order to prepare SHI-irradiated films, the as-prepared ESD TiO₂ films were used without sintering.

SHI was conducted using 15 UD Pelletron tandem accelerator facilities available in the Materials Science Beamline at the Inter-University Accelerator Centre (IUAC), New Delhi, India. The vacuum of the experimental chamber was in the range of 10⁻⁶ torr. The TiO₂ films, which act as blocking layers, were subjected to 100 MeV O ion irradiation with fluence of 1 × 10¹³ ions/cm². The electronic and nuclear energy loss values for 100 MeV O ions in TiO₂, calculated using the SRIM code simulation program (SRIM-2010) [24,25], were 1.284 × 10² and 6.739 × 10⁻² eV/Å, respectively. The range of O ions in this experiment is about 54.14 μm, indicating that the entire passage of ions in the film is dominated by electronic energy loss. Further experimental details were published elsewhere [26].

In order to compare the effect of the blocking layer, two kinds of DSSCs were assembled: (a) a pristine cell fabricated from the ESD TiO₂ blocking layer and (b) a SHI cell using an irradiated ESD TiO₂ blocking layer. In addition, a reference cell was fabricated from the TiO₂ blocking layer prepared by conventional spin coating (Ti (IV) bis (ethyl acetonato)-disopropoxide solution in 2 wt% of 1-butanol) and was also tested under identical conditions.
experimental conditions. Further, TiO2 photoanodes thickness about ~6 μm were prepared on the TiO2 blocking layer using TiO2 paste (Solaronix) by a doctor blade technique [27] and subsequently sintered at 450°C for 30 min in air.

N719 dye (di-tetrabutylammonium cis-bis(isothiocyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato)ruthenium(II)) was used to sensitize the TiO2 photo electrodes. The TiO2 electrodes were immersed overnight in a 0.3 mM dye solution containing a mixture of acetonitrile (ACN) and t-butyl alcohol (1:1 v/v) and dried at room temperature. A sandwich-type configuration was employed to measure the performance of the dye-sensitized solar cells, using a Pt-coated F-doped SnO2 film as a counter electrode and 0.5 M MPII (1-methyl-3-propylimidazolium iodide) with 0.05 M I2 in ACN as the electrolyte solution. Current–voltage characteristics of DSSCs were performed under 1 sun illumination (AM 1.5G, 100 mW cm⁻²) with a Newport (USA) solar simulator (300 W Xe source) and a Keithley 2,400 source meter (device area is 0.16 cm²). The different stages of the cell fabrication are schematically shown in Figure 1. Electrochemical impedance measurements were carried out using a potentiostat (IM6 ZAHNER) equipped with a frequency response analyzer (Thales) in the frequency range of 0.1 Hz–1,000 kHz. The results were analyzed with an equivalent circuit model for interpreting the characteristics of the DSSCs. Incident photon-to-current conversion efficiency (IPCE) of DSSCs was measured using PV Measurements Inc. (Model QEX7) with bias illumination with reference to the calibrated silicon diode.

The surface morphologies of the TiO2 thin films before and after SHI irradiation were studied by field-emission scanning electron microscopy (JEOL-JSM 6330F). The crystalline phases of the TiO2 films were determined by X-ray diffraction (XRD) using a diffractometer (Rigagu Denki Japan) with CuKα radiation. The conductivity of the samples was studied via the two-probe method.

**Results and Discussion**

Figure 2 shows the X-ray diffraction spectra of the ESD pristine and the SHI-irradiated TiO2 layers. Hereafter, the SHI-irradiated TiO2 layer is referred to as a layer formed by the ESD first and subsequently SHI-irradiated techniques. The characteristic peak observed at ~25.3° in both the films indicated the presence of an anatase phase of TiO2 (JCPDS 21-1272). The increase in the relative peak intensities observed in the SHI-irradiated sample shows that the SHI irradiation induced crystallization when compared to the as-prepared pristine ESD TiO2 films. The average grain size of the SHI-irradiated TiO2 films was found to be about 47 nm as estimated from Scherrer’s equation. The significant additional peak exhibited in the SHI-irradiated sample is not clearly understood.

Surface morphologies of the pristine and the SHI-irradiated TiO2 films are presented in Figure 3. The electrosprayed TiO2 films reveal an aggregation pattern, and the spherical particles form an interconnected porous framework of nano-sized building blocks (Figure 3). The observed nano-aggregated particles may be ascribed to the existence of a Coulombic force lower than the stretching force resulting from weak repulsion between adjacent spray droplets. Under SHI irradiation, these nano-aggregated TiO2 particles melted and solidified on the FTO substrate and consequently formed a rather
flat, nonporous structure with the FTO layer (see Figure 3). This results in a compact interface at FTO/TiO$_2$ for both blocking electron recombination and increasing electronic transport. The fragmentation of the aggregated particles into smaller grains under SHI irradiation can be explained by a thermal spike model. If a large amount of energy is deposited by the projectile ions to the electronic subsystem of the target material, this energy can be shared among electrons by electron–electron coupling and later transferred quickly to the surrounding lattice through electron–phonon coupling. Thus, a sudden temperature rise on the time scale of $10^{-12}$ s along the ion track resulted in a molten state. The subsequent heat transfer to the surrounding lattice results in resolidification of this molten liquid phase. If this cooling rate slows to a critical value, nucleation of crystalline phases can be expected along the ion trajectory [28,29]. Therefore, we speculate that the surface of the TiO$_2$ particles may undergo an ion-beam-induced molten state in a short duration of time ($10^{-12}$ s). These molten state particles were attached with FTO substrate, enhancing the inter-particle connectivity (compact) to improve the conductivity of the film. The measured conductivity of the pristine and the SHI-irradiated TiO$_2$ films found to be $2.31 \times 10^{-2}$ and 1.2 Scm$^{-1}$, respectively, indicating large improvement in the electron conductivity. Cross-sectional SEM images of the pristine and the SHI-irradiated TiO$_2$ films are illustrated in Figure 4.

Figure 4b suggests that the pristine ESD TiO$_2$ layer has nano-aggregates and an inhomogeneous interface (contact) with the FTO layer, mostly due to the removal of polymer templates from ESD coating during sintering treatment. The observed inhomogeneous TiO$_2$/FTO interface in the pristine sample was further compressed by SHI irradiation using O$_2$ ions. This interface modification was confirmed by Figure 4c, showing that the TiO$_2$ particles adhered well to the FTO layer. The thickness of the pristine film, ~1.1 μm, was reduced to ~0.67 μm after O ion irradiation. This is ascribed to the compact nature of TiO$_2$ film formed by SHI irradiation. It is noteworthy to mention that improving the compact nature of the TiO$_2$ blocking layer upon SHI irradiation can facilitate electron transport and also reduce electron recombination back to the electrolyte.

As shown in Figure 5, the ESD TiO$_2$ blocking layer DSSC (pristine cell) shows higher IPCE (maximum up to about ~53% at 530–540 nm) than the reference cell over the whole range of light wavelengths. This clearly demonstrates a ~16% improvement in external quantum efficiency from reducing the electron losses at
FTO/TiO$_2$ interfaces. It appears that the ESD is more efficient than the spin coating in terms of improving IPCE due to the formation of continuous films. Further, substantial improvement in IPCE was identified at lower wavelengths (380–420 nm), attributable to the SHI irradiation on the TiO$_2$ blocking layer. The IPCE can be rationalized using the following relation [30],

\[
\text{IPCE}(\lambda) = A \eta_{inj} \eta_{coll} 
\]

where $A$ is the absorptivity indicating the fraction of incident light absorbed by the dye molecules, $\eta_{inj}$ is the injection efficiency of dye molecules into the TiO$_2$ conduction band, and $\eta_{coll}$ is the collection efficiency. The parameters $A$ and $\eta_{inj}$ are directly related to dye loading on the TiO$_2$ surface. In the present work, we have controlled similar dye loading in the reference, pristine, and SHI-irradiated electrodes, as verified with a dye removal test using 1 M aqueous NaOH solution. Therefore, $A$ and $\eta_{inj}$ of all these samples can be treated to be equal, and the change in the IPCE is related to the improvement in $\eta_{coll}$. This improvement in $\eta_{coll}$ under SHI irradiation can be ascribed to (a) better adhesion of the TiO$_2$ blocking layer with the TCO substrate and (b) enhanced contact among TiO$_2$ particles. Hence, it is expected that the SHI-irradiated blocking layer may result in higher photoconversion efficiency.

Figure 6 shows the photocurrent density–voltage (J-V) characteristics measured under 1 sun (100 mW cm$^{-2}$ AM 1.5) and dark conditions. The photovoltaic parameters were estimated from Figure 6 and are summarized in Table 1. The photocurrent density ($J_{sc}$) was increased from 8.9 to 12.2 mA cm$^{-2}$, and the overall efficiency ($\eta$) was markedly improved from 3.8 to 5.1% by replacing the ESD TiO$_2$ compact layer, compared to the conventionally spin-coated blocking layer. This might be attributed to the highly compact nature of the ESD films, which provide more effective pathways for electrons. As a result, electrons can be collected faster at the TCO and transferred to the external circuit, resulting in improvement in the photovoltaic performance. However, there is no appreciable change in the open circuit voltage ($V_{oc}$) between these samples. When the ESD cell was treated with SHI irradiation, the open circuit voltage was further improved from 0.60 to 0.63 V, and consequently, the overall energy conversion efficiency improved from 5.1 to 5.5%. This may be because of the SHI irradiation, which melted TiO$_2$ particles and thereby improved electrical contact with the FTO substrate (denser and more compact) and among TiO$_2$ particles. This clearly demonstrates that the SHI irradiation enhances the blocking effect of electron recombination and creates a facilitating effect on electron transport.

A comparison of dark currents between the investigated cells provides qualitative information about the electron recombination process [31]. In DSSCs, preventing the recapture of photoinjected electrons by $I_3^-$ is vital to obtain a high open circuit photovoltage. By inserting the blocking layer between the FTO substrate and the TiO$_2$ mesoporous layer, the reaction possibilities of $I_3^-$ with the photoinjected electrons on the FTO substrate are significantly hindered, as demonstrated by the reduced dark current [31]. Here, the dark current–voltage curves of the DSSCs using different blocking layers are presented in the lower part of Figure 6. The less dark current observed in the SHI-irradiated cell

| Sample                          | $V_{oc}$ (V) | $J_{sc}$ (mA cm$^{-2}$) | F.F (%) | Efficiency (%) |
|---------------------------------|--------------|-------------------------|---------|----------------|
| Reference                       | 0.59         | 8.9                     | 71.9    | 3.8            |
| Pristine                        | 0.60         | 12.2                    | 69.3    | 5.1            |
| O$_2$ ion irradiated (1 × 10$^{13}$ ions/cm$^2$) | 0.63         | 12.3                    | 69.9    | 5.5            |

Table 1 Influence of TiO$_2$ blocking layer on photovoltaic parameters of DSSCs
compared with the pristine cell may be attributed to the better electrical contact between the blocking layer and the FTO substrate, and the compact nature of the blocking layer as well. Furthermore, during SHI irradiation, it is expected that Sn4+ particles from the FTO layer may fuse with the TiO2 layer occupying the oxygen vacancies in TiO2, thus lowering the Fermi level of TiO2. For instance, the Fermi level position of the Sn-doped TiO2 layer is lower than that of the TiO2 mesoporous layer, which is favorable for fast electron injection from mesoporous TiO2 particles to the conducting substrate [32].

Electrochemical impedance spectroscopy (EIS) provides valuable information on the kinetics of electron transport in the DSSCs with deeper understanding of the interfacial reactions at FTO/TiO2 [33] and therefore was employed to decipher the blocking layer effect in DSSCs. Figure 7 shows the Nyquist plots of the electrochemical impedance spectra. Their equivalent circuit is given as an inset in the figure. The charge transfer resistances RCT1 and RCT2 represent the resistances at the Pt/FTO and TiO2/dye/electrolyte interfaces, respectively. The electrochemical parameters were estimated by fitting experimental data with the equivalent circuit (inset of Figure 7) [34] and are summarized in Table 2.

The series resistance, Rs, was decreased markedly in the case of the pristine and O ion-irradiated electrodes, compared to the reference electrode. This is mostly associated with better electron transfer through the blocking layer due to better contact and better adhesion. The RCT2 value for SHI cells was increased markedly compared to the reference and the pristine electrodes. The increased RCT2 value may be mostly due to the fast electron transfer through the blocking layer. Hence, the increased electron transfer leads to lowering electron concentration of TiO2 mesoporous particles, which is responsible for observed high RCT2 (57.3 Ω) values in the O ion-irradiated sample.

The results described above suggest that contact among nanoparticles and the adhesion properties of a blocking layer with an FTO substrate may improve the performance of dye-sensitized solar cells. Further studies using different ion energies and fluence may further explain the role of electronic energy loss on these devices and allow development of precise control of the blocking layer.

**Conclusions**

An electrostatic spray deposition (ESD) technique followed by SHI irradiation using 100 MeV oxygen ions resulted in the formation of an efficient, dense TiO2 blocking layer between the TiO2 particle layer and the TCO substrate. The blocking layer promotes charge transport from the TiO2 layer to the TCO substrate by modifying the TCO/TiO2 interfaces and causes effective electrical contact between the two layers. The formation of an effective, compact blocking layer was possible due to instantaneous surface melting of the ESD TiO2 nanoparticles associated with a local temperature rise upon oxygen ion irradiation. Energy conversion efficiency was improved to a large extent (η = 5.5%), compared to that of the conventional blocking layer (η = 3.8%), mainly due to the increase in electron transport through the blocking layer, resulting from better contact among TiO2 nanoparticles and better adhesion with the TCO substrate.

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**Table 2** Influence of TiO2 blocking layer on electrochemical parameters of DSSCs

| Sample                                | Rs (Ω) | RCT1 (Ω) | RCT2 (Ω) |
|---------------------------------------|--------|----------|----------|
| Reference                             | 20.3   | 6.7      | 41.9     |
| Pristine                              | 13.6   | 8.6      | 39.8     |
| O2 ion-irradiated (1 × 1013 ions/cm²) | 13.9   | 7.9      | 57.3     |

**Figure 7** Nyquist spectra (measured under light illumination (100 mW cm⁻²)) of DSSCs. The inset represents the impedance spectra expanded in the high frequency ranges. The scattered points are experimental data, and the solid lines are the fitting curves.
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