Low temperature chemically sintered nano-crystalline TiO$_2$ electrodes for flexible dye-sensitized solar cells

H.C. Weerasinghe [a], P.M. Sirimanne [a], G.V. Franks [b], G.P. Simon [a], Y.B. Cheng [a,*]

[a] Department of Materials Engineering, Monash University, Wellington Road, Clayton, Vic 3800, Australia
[b] Department of Chemical and Biomolecular Engineering, University of Melbourne, Vic 3010, Australia

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ABSTRACT

Chemically sintered, mesoporous TiO$_2$ electrodes with improved interparticle contact and bond strength were prepared on indium tin oxide (ITO) coated plastic substrates in the absence of any organic binders, using hydrochloric acid as the sole reagent to encourage interparticle connectivity. The degree of chemical sintering as a function of the HCl concentration was evaluated using a newly developed nanoscratch technique. The rheological properties of the titania paste was influenced by the HCl, as was the resultant bonding of the TiO$_2$ grains both to each other and to the substrate. The mechanism for the low temperature chemical sintering could be due to the surface modification of titania particles during the HCl treatment and subsequent improved chemical bonding at particle contact points. The improved strength and chemical bonding of the particle network led to improved photovoltaic properties of the resulting solar cells. The highest value of light to electrical energy conversion efficiency obtained was 5% for the plastic-based dye-sensitized solar cells, under 1 sun (100 mW cm^{-2}). This thus represents a novel method for ensuring the appropriate titania nanomorphology using room temperature treatment on a flexible substrate, as compared to the required thermal treatments in excess of 400 °C normally required in conventional ITO glass-based dye-sensitized solar cell devices.

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1. Introduction

Glass-based dye-sensitized solar cells (DSCs) have been extensively studied due to their potential for manufacturing low cost solar cells and over 11% conversion efficiencies, compared to that of silicon solar cells [1,2]. In most of these studies, titania electrodes are usually prepared on conductive glass substrates from screen-printable titania paste containing organic binders, followed by an annealing process at 450–500 °C. The sintering process is essential to achieve better electrical contact between the semiconductor films and the conducting layer on the substrates, as well as between TiO$_2$ particles where interparticle necking is induced. Another purpose of the sintering is to remove the organic binder by decomposition. Increasingly, the requirement and desire is for such solar cells being made on flexible, plastic substrates which are light weight, can be bent and printable, and are more suitable as small-scale power generation units in a modular fashion. However, the temperature limits of plastic substrates will usually preclude such sintering processes and the use of organic binders for plastic-based solar cells. Any resultant poor adhesion between

particle–particle and particle–substrate in such plastic solar cells leads to lower power conversion efficiencies for the flexible cells, compared to rigid, glass-based DSCs. Alternative treatments are thus required in order to improve the connectivity between TiO$_2$ particles as well as between the film and the conductively coated plastic substrate. To date, it has been reported that a plastic-based dye-sensitized solar cell can be prepared by using an interparticle connecting agent in the TiO$_2$ paste and that a maximum power conversion efficiency of 6.4% has been achieved [3]. The efficiency of these cells is higher than those prepared from other techniques used to produce DSCs on plastic substrates such as the lift-off technique [4], low-temperature heating [5], microwave irradiation [6], hydrothermal crystallization [7,8] chemical vapour deposition [9], mechanical compression [10] and electrophoretic deposition [8,11]. Most of these latter techniques need an additional film processing step during the electrode preparation, or are expensive and required complex processes which are difficult to replicate in industry. Indeed, a roll-to-roll printing process of titania films is considered as the most suitable and desirable technique for mass production of flexible solar cells. Preparation of binder-free titania pastes that have a suitably high viscosity and better adhesion to the hydrophobic indium tin oxide (ITO) surface of the plastic substrates used in roll-to-roll printing has always been a challenge and only a few such titania pastes have been developed [3,12].

* Corresponding author. Tel.: +61 3 99054930.
 E-mail address: Yibing.Cheng@eng.monash.edu.au (Y.B. Cheng).

In this report, we propose a single-step coating method for the preparation of TiO$_2$ electrodes using readily available P-25 (Degussa) titania powder with improved connectivity between the titania particles, and between the titania film and the ITO layer on the polymer substrate, achieved by introducing hydrochloric acid as an additive during the film deposition. The enhancement in both photovoltaic and mechanical properties of these electrodes is demonstrated in this work.

2. Experimental

2.1. Preparation of nanoparticulate TiO$_2$ electrodes

Titania suspensions of 30 wt% in ethanol (3 g of P-25 TiO$_2$ powder dispersed in 7 g of ethanol) were prepared by ball milling [13], with the suspensions being chemically modified to form pastes by the inclusion of different volumes of a 1 M HCl aqueous solution to the slurry, followed by hand milling using an agate mortar and pestle. Slurries were prepared with different amounts of the aqueous 1 M HCl solution and their viscosity found to be highly dependent on acid amounts. The magnitude of the viscosity of TiO$_2$ suspensions with different concentrations of HCl was studied using an AR-G2 rheometer (TA Instruments) at different shear rates. TiO$_2$ films were prepared, respectively, on glass, polyethylene naphthalate (PEN) and ITO coated PEN (known as ITO|PEN, Peccell Technologies, Inc., Japan) substrates by the doctor blade technique. The TiO$_2$ films were allowed to dry at room temperature for 10 min. Electrodes produced on PEN substrates were post-heat-treated on a hot plate at 150 °C for another 30 min, whereas the electrodes on glass substrates were sintered at 500 °C for 30 min.

2.2. Mechanical properties of nanoparticulate TiO$_2$ electrodes

As an indirect means to assess the interparticle connection, mechanical properties of nanoporous TiO$_2$ electrodes were evaluated using a Hysitron Nanoindenter by a method we recently reported [13]. Three-sided pyramidal Berkovich standard tip (angle of 142.35°, half-angle 65.35°, aspect ratio of the tip is 1:8, radius of curvature approximately 150 nm) was used for scratch formation of the different TiO$_2$ films. A number of well spaced, distinct nanoscratches of a fixed size (20 μm in length and 2 μm in depth) were produced under the displacement control mode on the surface of the TiO$_2$ films and the normal and lateral forces acting on the indenter were measured. The surface and cross-sectional morphologies and the elemental composition of the electrodes were analysed using FEI Nova NanoSEM with an EDAX X-ray detector and a VG 310F Scanning Auger Microscope (Nanoprobe). For the cross-sectional analysis, a mold was prepared by imbedding the TiO$_2$|PEN electrodes in a resin (Epofix), allowing to dry at the room temperature and then to cure in an oven at 60 °C. The mold was subsequently ground by hand on a SiC sand paper, and diamond polished using a mechanical polisher before imaging.

2.3. Preparation of solar cell devices

TiO$_2$ coated ITO–PEN electrodes were sensitized with cis-bis (isothiocyanato)bis(2,2′-bipyridyl-4,4′-dicarboxylato)-ruthenium(II)-bis-tetrabutyl ammonium N719 dye (Solaronix SA, Switzerland) by dipping in a 0.2 mM solution of the dye in a mixed solvent of acetonitrile and ethanol (1:1) for around 4 h. Dye coated films were rinsed with pure ethanol and dried at room temperature. Fully plastic dye-sensitized solar cells were fabricated by sandwiching catalytic Pt|ITO|PEN films (Peccell Technologies, Inc., Japan) with the dye coated TiO$_2$|ITO|PEN electrode using a 25 μm thick thermoplastic hot-melt sealing foil as a spacer (Solaronix SA, Switzerland). The electrolyte was comprised of a mixture

of 0.04 M iodine, 0.40 M 4-tert-butylpyridine, 0.40 M lithium iodide, 0.30 M N-methylbenzimidazole (in acetonitrile and 3-methoxypropionitrile by volume 1:1) and was filled into the cavity space by capillary action. Current–voltage characteristics (I–V) and incident photon-to-current conversion efficiency (IPCE) of the cell with the configuration of Pt|electrolyte|N719|TiO$_2$ were studied under irradiation of white light (100 mW cm^{-2}) and monochromatic light using an Oriel solar simulator system. The interfacial electrical properties of the cells were studied by applying −0.4 V on the working electrode under a two-electrode configuration, at the dark condition, with the same electrolyte, using a multi-channelled potentiostat (Princeton Applied Research) coupled with a computer. The charge transfer resistance at the electrolyte–electrode interface was evaluated using EClab software.

3. Results and discussion

3.1. Slurry rheology and interparticle interactions

The change in the appearance of the TiO$_2$ slurry (a) before and (b) after adding HCl is shown in Fig. 1. The milled 30 wt% titania suspension appeared to be a low viscosity liquid in pure ethanol (Fig. 1A (a)). The viscosity of the TiO$_2$ suspension was found to increase when HCl was added, and it became a paste after the addition of 100 μl of 1 M aqueous HCl solution in to 4 ml of the ethanol based titania suspension (Fig. 1A (b)). The variation in paste rheology was also studied using rheometry as a function of shear rate and the results are shown in Fig. 2. The rheology property of the suspension is an important issue associated with printing. The pure ethanol suspension with low viscosity did not produce good quality thin films. On the other hand, the shear thinning viscosity (paste-like behaviour) that resulted when the acid was added produced a suspension resulting in high quality thin films of TiO$_2$ nanoparticles.

The viscosities of suspensions are related to the interparticle interaction forces [14,15]. The forces acting between the particles in the TiO$_2$ suspensions are the electrical double layer repulsion and van der Waals attraction [16,17]. The viscosity change observed in the slurries is possibly due to the reduction of double layer repulsion with the introduction of HCl which increases in the electrolyte concentration in the slurry. Thus, van der Wals attraction can become dominant in such a system increasing the interparticle attraction and hence the viscosity of the slurry.

Interparticle connection in the titania film is formed during drying by dehydration of hydrogen bonded network of TiO$_2$ particles covered with hydroxyl groups as explained in Fig. 1B [3]. It has been shown that the hydroxyl content on titania surface considerably increased due to the HCl treatment [18]. Therefore interparticle bonding of the electrode could be enhanced by the HCl treatment which increases the amount of hydroxyl groups on the titania surface. The improved interparticle connection may also be a result of a chemical reaction between HCl and TiO$_2$. However, there is not enough evidence in the current study to verify this assumption.

3.2. Morphology and the mechanical stability of the TiO$_2$ films

Thin titania films of ∼1 μm were prepared on ITO/PEN films by slurries with and without HCl (the HCl concentration in the slurry was 0.05 M) (Fig. 3). The morphology of TiO$_2$ particles can be clearly identified in the film containing no HCl (Fig. 3a). In the 1 μm thick film prepared by the slurry containing HCl, however, TiO$_2$ particles appear as clusters, together with becoming more embedded in the ITO surface (Fig. 3b). This effect is likely due to the slight dissolution of the ITO surface in the wet acidic slurry, followed by re-precipitation around TiO$_2$ particles closer to the TiO$_2$|ITO
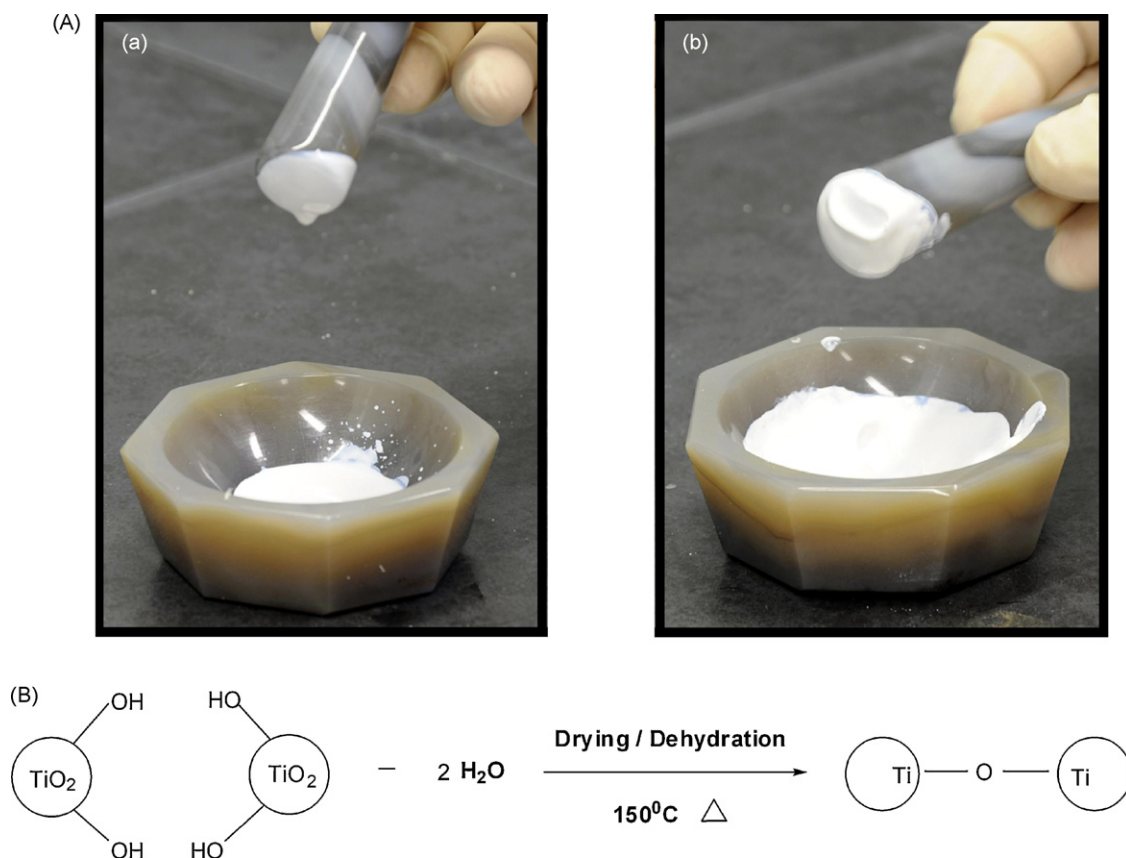
Fig. 1. (A) Comparative images of change of viscosity of the TiO$_2$ colloid (a) before and (b) after adding HCl (0.025 M). (B) Formation of Ti–O–Ti bonds among the TiO$_2$ particles.

interface during the drying of the film. For a better understanding, surface elemental analysis of the titania films was carried out using Scanning Auger Microscopy (SAM). A higher indium to titanium ratio (28:72) was observed for the acid-treated film, compared to the non-treated film (3:97). This result indicates that the surface of the TiO$_2$ particles close to the ITO layer in the acid-treated film is partially coated by ITO. The effect of HCl in the slurry of 0.05 M HCl on thicker TiO$_2$ films (∼8 μm) was also investigated. A similar morphology to that of Fig. 3a was observed for thicker titania electrodes prepared using both acid treated and non-acid treated slurries. Although HCl in the acidic titania slurry reacted with the

Fig. 2. Change of viscosity of the TiO$_2$ colloids with the concentration of HCl against the sheer rate for (a) non-acidic, (b) 0.025 M, (c) 0.05 M and (d) 0.1 M HCl systems.

ITO layer and produced a coating on the TiO$_2$ particles near the TiO$_2$/ITO interface, no distinguishable change was observed from SEM on the surface of the acid-treated thick (∼8 μm) TiO$_2$ electrodes. This indicates that the clusters are formed only close to the ITO–TiO$_2$ interface but not through the entire TiO$_2$ film.

Scanning electron micrographs of the cross-section of titania films prepared from (a) acid treated and (b) non-acid treated slurries on ITO|PEN substrates are shown in Fig. 4, where the specimens were imbedded in a resin and cured at 60 °C. As can be seen from the SEM images, good adhesion of the TiO$_2$ film to the ITO|PEN substrate exists in the specimen prepared by the HCl-containing titania slurry (Fig. 4a). A gap arises between the TiO$_2$ film and the underlying ITO layer in the specimen prepared without HCl (Fig. 4b), which can be attributed to the thermal stresses developed during drying and curing of the resin for the SEM sample preparation. The good adhesion observed in the film with HCl (Fig. 4a), indicating that the bonding between the TiO$_2$ film and the substrate was greater than in the absence of acid, since the same thermal stresses would be expected in both cases. The results suggest that the addition of HCl has enhanced adhesion of the TiO$_2$ film and the ITO|PEN substrate. EDX profiles across the acid treated and non-acid treated titania films are also shown in Fig. 4. A significant degree of diffusion of indium was observed in the first few microns of the TiO$_2$ film prepared by the acid-treated slurry.

A large contact area and strong bond between individual TiO$_2$ particles is important, because good connectivity of the TiO$_2$ particle network is likely to result in good electrical conductivity of the particle network and thus efficient photon to electron conversion. The bonding strength between particles can be estimated from macroscopic measurement of the mechanical integrity of the thin film and previously reported nano-indentation technique [13] was used to evaluate the bonding strength between the particles. The
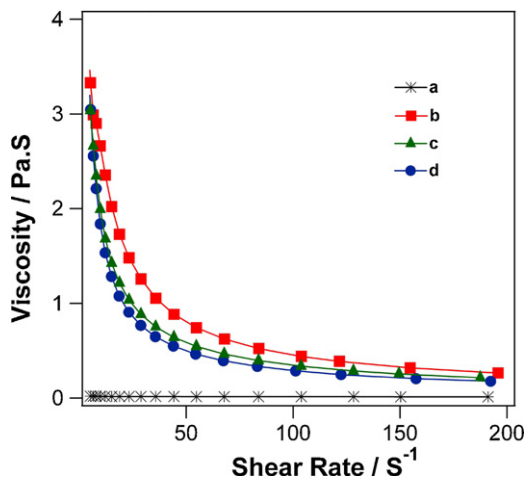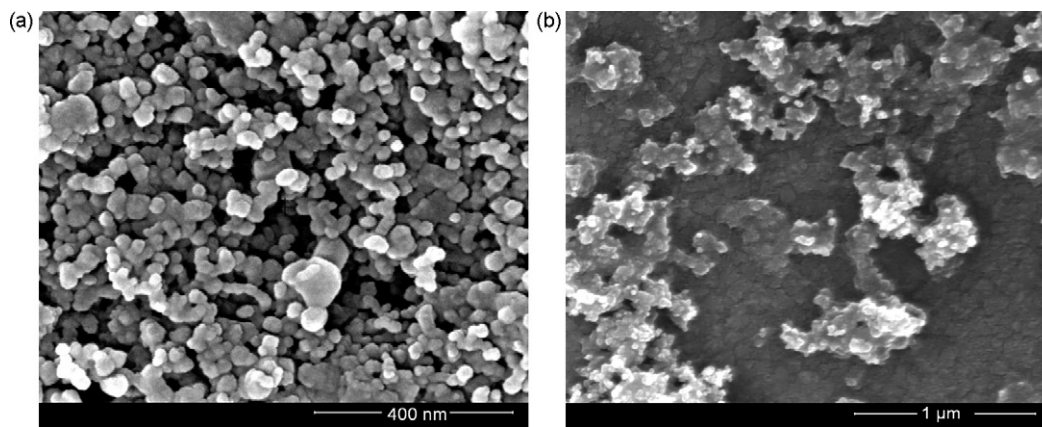
Fig. 3. SEM images of the TiO$_2$ films prepared by using (a) non-hydrochloric and (b) 0.025 M acidic titania slurries, thickness of the electrode ~1 μm.

measurements were carried out for TiO$_2$ films prepared on PEN, ITO|PEN and FTO|glass substrates, respectively. The variations of normal and lateral forces against the scratch distance (20 μm in total) for the electrodes prepared on the PEN substrate (without the ITO coating) are shown in Fig. 5A and B. A gradual enhancement in both normal and lateral forces was observed with increasing of the acidity of the titania slurry. A similar variation trend for both lateral and normal forces was also observed for the TiO$_2$ films prepared on ITO|PEN substrates by using titania slurries containing HCl up to 0.05 M [Fig. 5C and D]. However, a dramatic increase of normal and lateral forces is observed for the titania film prepared on the ITO|PEN substrate containing 0.1 M HCl concentration [curve d in Fig. 5C and D]. EDX analysis on the surface of the titania film confirmed the diffusion of indium to the top surface of the electrode prepared using titania slurry containing 0.1 M HCl concentration. This result suggested that the formation of TiO$_2$ clusters due to ITO diffusion could occur up to the top surface of the titania electrode when the HCl concentration in a TiO$_2$ slurry was high. A top surface morphology of such a film in Fig. 6 showed the for-

mation of In$_2$O$_3$–TiO$_2$ clusters. It can therefore be speculated that a stronger bonding should exist at the TiO$_2$–ITO interface for the films prepared with lower acidic concentrations (Fig. 6). It was also of interest to compare the strength of the particle network (and thus interparticulate connectivity and bonding) achieved though hydrochloric treatment with that obtained from conventional high temperature sintering. Fig. 5E and F shows the forces exerted on the indenter during nanoscratching of non-acid containing titania films prepared on plain glass substrates heated at (a) 150 °C and (b) 500 °C. Both normal and lateral forces obtained for the titania film sintered at 500 °C were comparable to the values obtained for the films prepared using titania slurry containing 0.05 M HCl, clearly indicating that the particle–particle bonding and network connectivity in the TiO$_2$ film were enhanced due to the hydrochloric treatment.

The information from SEM, EDX and SAM analyses also indicate that a small amount of ITO dissolves in the HCl diffused into the titania film near the ITO–TiO$_2$ interface. During drying, the dissolved ITO will precipitate out as a solid phase assisting the adhesion of the
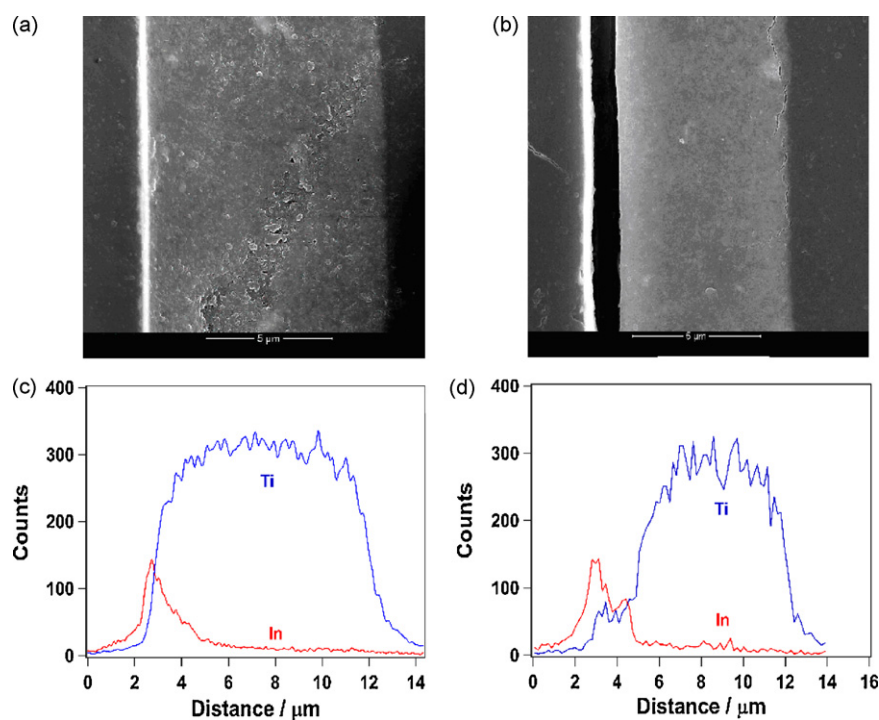

Fig. 4. Cross-section scanning electron micrographs of titania films prepared from (a) acid treated (0.025 M) and (b) non-acid treated slurries and EDX profiles across (c) acid treated and (d) non-acid treated titania films.
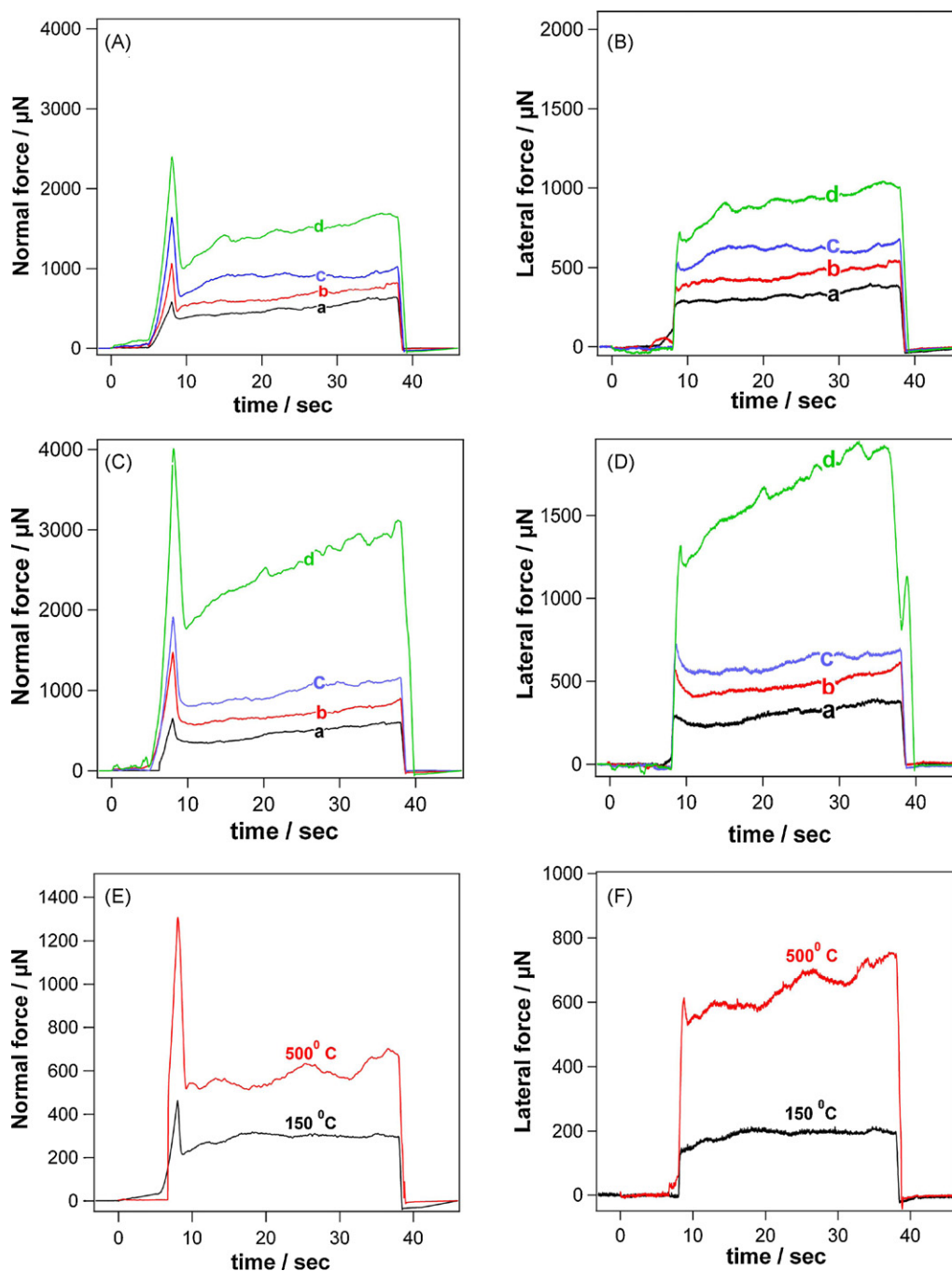
Fig. 5. Variations of (A) normal and (B) lateral forces against displacement in nanoscratch tests for the TiO$_2$ electrodes prepared on the PEN substrate; (C) normal and (D) normal forces in the same tests for the electrodes prepared on the ITO|PEN substrate. The HCl concentration in the titania slurries was (a) zero, (b) 0.025 M, (c) 0.05 M, (d) 0.1 M respectively. Variations of (E) normal and (F) lateral forces for TiO$_2$ films prepared using a non-acid treated TiO$_2$ slurry on plain glass heat-treated at 150 °C and 500 °C respectively.

TiO$_2$ film onto the ITO|PEN substrate, which is schematically illustrated in Fig. 7. Mechanical stability of these electrodes on flexible substrates were also tested by the bending deformation and the HCl treated electrodes showed much improved connectivity of the titania film with the substrate compared to the non-treated films.

3.3. Photoelectrical properties and Impedance spectroscopy of solar cells

IPCE spectra of two different cells are shown in Fig. 8 for TiO$_2$ films prepared using (a) non-acidic slurry and (b) TiO$_2$ slurry with HCl concentration of 0.025 M on ITO–PEN substrates. The maximum

IPCEs of 43% and 47% at 525 nm were observed respectively for the untreated and treated cells. The corresponding current–voltage characteristics of non-treated and HCl treated cells are shown in Fig. 9 and the results summarized in Table 1. The short cir-

Table 1
Performance of the electrolyte|dye|TiO$_2$ cells.

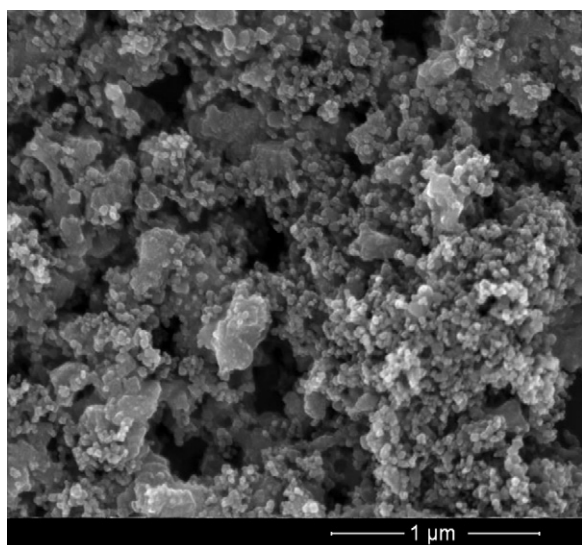| TiO$_2$ slurry	J_{sc} (mA cm^{-2})	V_{oc} (V)	ff	η (%)	R_0 (Ω)	R_1 (Ω)	R_2 (Ω)
Non-treated	9.79	0.663	63	4.05	15.7	7.1	15.5
0.025 M acidic	10.03	0.735	69	5.00	17.4	7.0	12.0
0.05 M acidic	9.29	0.671	59	3.67	22.7	5.2	10.1

Fig. 6. Top surface morphology of the TiO_2 film prepared using 0.1 M acidic titania slurry, thickness of the electrode ~8 μm.
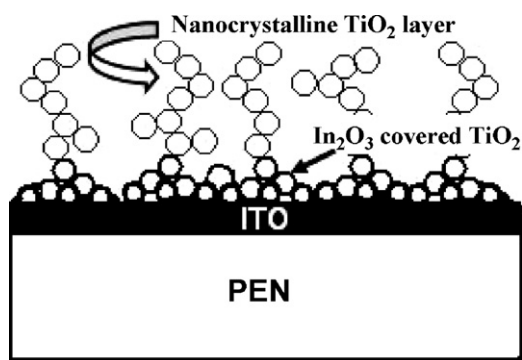

Fig. 7. Schematic illustration of TiO_2 nano-crystalline film prepared using hydrochloric treated titania slurry.
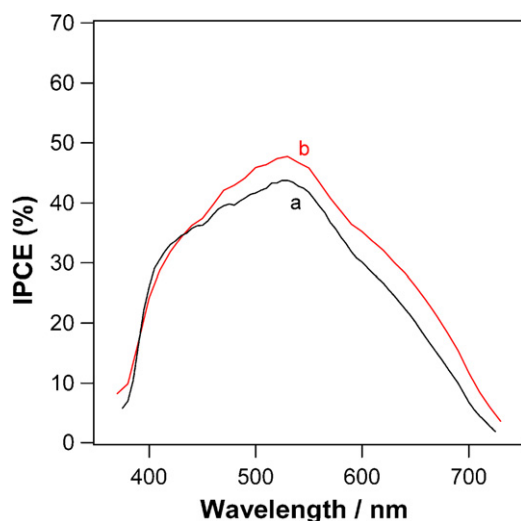

Fig. 8. The IPCE spectra for cells prepared by using (a) non-acidic and (b) 0.025 M HCL TiO_2 slurries.

Fig. 9. Current–voltage characteristics (under illumination) of Pt|electrolyte|N719|TiO_2 cells prepared using titania slurries with different HCl concentrations (a) non-acid treated (b) 0.025 M. Current–voltage characteristics of solar cells at the dark also shown in similar colour codes (c, d). The insert shows the variation of the photo-conversion efficiency (η) with the HCl concentration of the TiO2 slurry.

cuit photo-current was found to increase slightly for the 0.025 M treated cell, but a noticeable improvement for the open circuit voltage and fill factor was observed. The maximum efficiency of 4.05% was observed for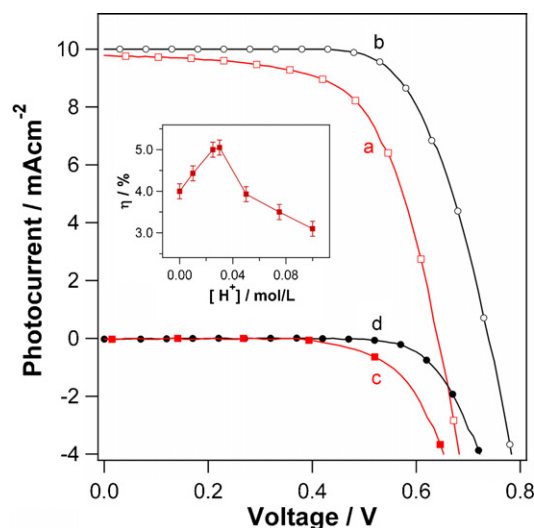 non-treated electrodes. The maximum overall light-to-electricity conversion efficiency of 5% was achieved for the electrodes prepared using the 0.025 M acidic slurry. The enhancement of the efficiency is mainly due to the increment of the photo-voltage and the fill-factor. The improved connectivity of the TiO_2 particle network as well as the bonding of the film to the substrate due to the acid treatment has reduced the charge recombination in the TiO_2 film. However significant photo-current increment is not observed for the HCl treated electrodes, possibly due to the damage of the ITO conducting layer on the PEN substrate. The performance of the solar cells was found to deteriorate for the slurries above 0.05 M hydrochloric concentration. The variation of efficiencies with increasing acid concentration of the titania slurry is shown in the insert in Fig. 9. Optimum efficiency is reported for the solar cells fabricated using slurries with ~0.025 M HCl concentration and higher or lower concentrations were found to be less optimal.

Electrical impedance spectroscopy is a useful technique to examine the interfacial electrical properties of DSCs [19]. Internal resistances of the TiO_2 film and the substrate/film interface were studied using electrical impedance spectroscopy. Three semicircles usually obtained in the Nyquist diagram that in the order of increasing frequency are attributed to the diffusion within the electrolyte, the electron transfer at the oxide/electrolyte interface and the redox reaction at the platinum counter-electrode [12,20,21]. The intercept with the real axis at high-frequency range corresponds to the contact resistance between the TiO_2 and the transparent conducting oxide or the ohmic resistance of the cell [22]. Impedance spectra for the electrodes prepared using titania slurries with different HCl concentrations (a) non-acid treated, (b) 0.025 M and (c) 0.05 M are shown in Fig. 10. For this work, only two typical distinct semicircles are seen as observed by others [23]. Nyquist plots and the equivalent circuit for the cell is shown in the insert of Fig. 10. Semicircle corresponding to high range of frequencies (left semicircle with diameter of R_1) and middle range of frequencies (right semicircle with diameter of R_2) respectively represent the redox reaction at the platinum counter-electrode and the electron transfer at the oxide/electrolyte interface, electron conduction in the electrode as well as charge injection to the electrode. The values of R_0, R_1 and R_2 are also summarized in Table 1. The Nyquist plots were found to shift to the right, i.e. an increase of R_0, with increas-
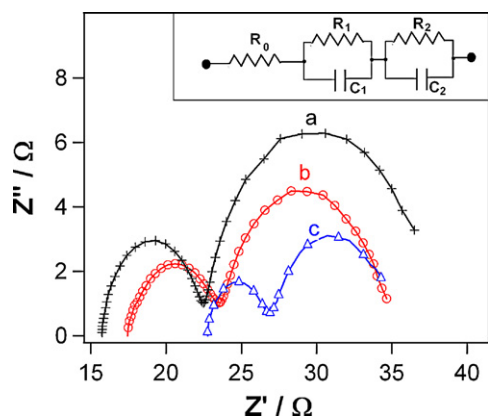
Fig. 10. Impedance spectra of Pt|electrolyte|N719|TiO$_2$ cells prepared using titania slurries with different HCl concentration (a) non-acid treated (b) 0.025 M and (c) 0.05 M.

ing acid concentration of the titania slurries, indicating an increase in the sheet resistance of the ITO–PEN substrate due to the dissolution of the ITO layer. The reduction of R_2 with the increase of acid concentration for treated electrodes provided further evidence for the improved particle–particle as well as the film–substrate connectivity.

4. Conclusions

A highly viscous binder-free, P-25 titania paste was developed by adding an appropriate concentration (0.025 M) of HCl into the ethanol based titania slurry. Mechanically stable, thick, porous nano-crystalline TiO$_2$ films with much improved adhesion to the substrate also resulted. Improved adhesion of the film with the substrate was found to be due to the slight dissolution of the ITO layer during the spreading of the wet slurry on the ITO–PEN. EDX profiles across the acid treated film showed some diffusion of indium within the first few microns of the film. A nanoscratch technique was used to evaluate the mechanical properties of the nanoporous TiO$_2$ films, which could be correlated to the interparticle contacts in the films. HCl treatment was found to improve the bonding between titania particles and the strength of the film was found to increase with the increase of the HCl amount. Titania electrodes prepared by using P-25 titania slurries with the acid concentration of 0.025 M exhibit higher performance in flexible-DSCs. The maximum power conversion efficiency of 5% has been achieved under 1 sun conditions.

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