



A seeding method to change primary particle of oriented attachment network titanium dioxide for dye-sensitized solar cells



Ping-Lin Kuo*, Chun-Hou Liao

Department of Chemical Engineering, National Cheng Kung University, Tainan 70101, Taiwan, ROC

HIGHLIGHTS

- The seeding method was used to improve thermal stability and thermal stress.
- The OA S2 has high surface area, lower R_w and perfectly aligned lattice.
- A higher efficiency was achieved by OA S2 as compared with commercial JGC 18NRT.

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ABSTRACT

In this paper, we use seeding methods to improve crystal thermal stability and thermal stress by oriented attachment (OA) particles as seeds. The OA S2 synthesized by repeating two times the seed process has pure anatase phase even if was calcined and high surface area ($113.2 \text{ m}^2 \text{ g}^{-1}$). Further, the lattice images of OA S2 obtains from oriented attachment mechanism showed perfect alignment in grain orientation and no grain boundary appears between the necking particles. The films of OA S2 after calcination at $500 \text{ }^\circ\text{C}$ is homogeneous without cavities over large area. The photovoltaic performance of dye-sensitized solar cells made of OA S2 exhibits higher J_{SC} and FF than the devices made of JGC 18NRT. The reason for the higher J_{SC} can be attributed to the high surface area and pure anatase phase. The OA S2 shows the lower R_w (charge transport resistance) than the JGC 18NRT. It is confirmed that the OA S2 film has the capability of higher electron transmission due to oriented attachment structure, so it displays low internal resistance and results in higher FF. A higher light-to-electricity power conversion efficiency of 6.10% is achieved by applying the OA S2 as compared with JGC 18NRT (5.85%).

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1. Introduction

The dye-sensitized solar cell (DSSC) is one of the most promising low-cost, high-efficiency, and environmentally friendly solar cells [1]. The DSSC is composed of the high-surface-area TiO_2 film on a conducting glass substrate, high absorption efficiency sensitized-dye, I^-/I_3^- redox electrolyte solution, and counter electrode with platinum coating. The dynamics competition process in DSSC for the initial events of electron injection and dye regeneration leading to photo-induced charge separation, which occurs in the femto-second to nanosecond time scale. However, the electron transport in the TiO_2 film and interfacial electron recombination on the TiO_2 surface with I_3^- are in milliseconds or even seconds domain [2].

Therefore electron transport and interfacial recombination are the key factors affecting the efficiency.

In order to accelerate electron transport and decrease interfacial recombination, 1D nanostructure TiO_2 (nanotubes [3–5], nanorods [6], and nanowires [7,8]) are prepared for DSCs. It was anticipated that they would have a single crystalline structure to facilitate incremental electron transportation in the photoelectrode and less surface defects for reducing interfacial recombination on the TiO_2 surface. However, compared with nanoparticles, 1D structures possess lower surface-area that affects dye adsorption and then decrease current density and conversion efficiency.

In order to maintain high surface-area for increasing electron transport and decreasing interfacial recombination, the Adachi's group synthesizes the network structure of single-crystal-like TiO_2 [9]. The TiO_2 grow by the "oriented attachment" mechanism resulted in that the orientations of crystals of fused nanoparticles with completely aligned orientation. The OA TiO_2 possess high

* Corresponding author.

E-mail address: plkuo@mail.ncku.edu.tw (P.-L. Kuo).

surface-area due to small particle size (2–5 nm) and accelerate electron transport as a result of perfectly aligned lattice. A high light-to-electricity power conversion yield of 9.3% was achieved by applying OA as the titania thin film of dye-sensitized solar cells. But the OA TiO₂ exists two problem: (1) after calcination at 500 °C the anatase phase TiO₂ change to rutile phase; (2) the OA film obtained from terpineol base paste is easy to crack after calcination.

The seeding method is easy method to change material morphology, Kuang group use seeding method to increase TiO₂ nanowire arrays surface area [10,11]. In this paper, we use seeding methods to improve crystal thermal stability and thermal stress resulted from the increased particle size. After calcination the OA S2 film present pure anatase phase and on crack over large area. An enhancement of conversion efficiency based on that the OA S2 (6.10%) is achieved as compared with JGC 18NRT (5.85%).

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2. Experimental

2.1. Preparation of oriented attachment TiO₂

Commercially available titanium isopropoxide (TIPO, 97%) and acetylacetone (AA, 99.8%) were used as received. The oriented attachment TiO₂ was synthesized according to the work of Adachi's group [9]. TIPO was mixed with AA and adjusted to pH 2, then stirred one day at 50 °C. The final solution was placed in a Teflon-lined autoclave and aged at 120 °C for three days. The network TiO₂ was designated as OA. The OA TiO₂ as seed particles again was mixed the precursor-solution and then placed in a Teflon-lined autoclave, lastly aged at 120 °C for three days. The network TiO₂ was designated as OA S1. The OA S2 and OA S3 were synthesized by repeating the seed process.

2.2. Device fabrication

For the preparation of the nanoporous TiO₂ layers with OA and OA S1–S3, the viscous slurries were prepared by the following procedure [12]. The TiO₂ was dispersed in ethanol and then mixed with terpineol and ethyl cellulose. After removing the ethanol with a rotary-evaporator, the final paste was obtained. The FTO glass plates were cleaned by surfactant, acetone and isopropanol and subsequently immersed into a 40 mM TiCl₄ aqueous solution at 70 °C for 30 min, lastly washed with water. Then the OA and OA S1–S3 TiO₂ paste and commercial paste JGC 18NRT (JGC Catalysts and Chemicals Ltd., Japan.) were coated onto the FTO substrate using the screen-printing method. The coated films were baked at 150 °C for 30 min and then sintered at 450 °C for 15 min and at 500 °C for 15 min. After calcination, the TiO₂ film was treated with 40 mM TiCl₄ solution as described above. The films were sintered again at 450 °C for 30 min. After cooling down to 80 °C, the TiO₂ electrodes were sensitized by immersing into dye solutions of 0.5 mM N719 dye (Solaronix) in acetonitrile and tertiary butanol (in the volume ratio of 1:1) at room temperature for 16 h. The dye-covered TiO₂ films and the Pt counter electrodes were assembled into sealed sandwich-type cells by heating with hot melt sealing foil (SX1170-25, 25 μm thickness, Solaronix) used as spacers between the electrodes. A hole was made by a sandblasting drill on the counter electrode, allowing the internal space between the two electrodes to be filled with electrolyte solution (0.1 M LiI, 0.6 M DMPH, 0.05 M I₂, and 0.5 M TBP in 3-Methoxypropionitrile, MPN)

using a vacuum backfilling system. After electrolyte filling, the hole was sealed with a thin glass sheet.

2.3. TiO₂ characterization and photovoltaic

Characterization of the TiO₂ are performed with transmission electron microscopy (TEM) at 80 kV (JEOL, JEM-1200EX), high resolution-transmission electron microscopy (HR-TEM) (FEI Tecnai G2), X-ray diffraction (XRD) at 40 kV and 30 mA (Rigaku, RINT2000), and isotherm of nitrogen adsorption at 77 K (Micromerit ASAP 2020), to get TiO₂ particles size, crystalline structures, surface area and pore size distribution respectively. Characterizations of the TiO₂ electrodes are measured by field emission scanning electron microscope (FE-SEM) at 10 kV (JEOL, JSM-6700F) to get TiO₂ films morphology.

Photocurrent–voltage measurements were performed using an solar simulator (SAN-EL, Model:XES-301S, Japan). The electrochemical impedance spectroscopy (EIS) measurements in this study were carried out by applying bias modulation near the open circuit condition (Voc) [13]. EIS measurements of cells were recorded in a frequency range from 0.05 Hz to 1 MHz with an ac amplitude of 10 mV. The active area of the dye-coated TiO₂ film was 0.16 cm².

3. Results and discussion

In this study, the system used acetylacetone (AA) not only as a stabilizer for TIPO to reduce the hydrolysis rate of the alkoxide, but also as a shape controller to specifically adsorb on the particle surfaces. In accordance with Adachi's group, the TiO₂ has characteristic crystal growth by the oriented attachment mechanism and forms a network of single-crystal-like anatase nanowires [9]. The network TiO₂ (designated as OA) was composed of fused nanoparticles with diameters of only 3–5 nm as shown in Fig. 1(a). Fig. 1(b) presents the SEM image of surface morphology OA film obtained from coating of terpineol base paste, followed by calcination at 500 °C for 30 min. The high-magnification SEM image (Fig. 1(b) inset) demonstrates that most of the particles are connected with each other and form a network of nanoparticles. However, the OA film of the low-magnification SEM image showed cracks due to the thermal stress after calcinations. In addition, the XRD pattern of the OA particle shows the existence of anatase and rutile phase as a result of calcinations (Fig. 1(c)).

Seeding methods reacting at 120 °C were used to improve crystal thermal stability and to release thermal stress by OA particles as seeds. Fig. 2(b)–(d) shows the TEM images of TiO₂ with three different seeding times, labeled as OA S1, OA S2, and OA S3. The OA S1–S3 seem to be the network structure with coating grown on the OA particles. As the seeding times increased, the network TiO₂ particle size increased (Fig. 2(b)–(d)). On the other hand, in order to understand the effect of the growth temperature on the morphology of the seeding particles, the different seeding temperature are compared in Fig. 2 (b) and (e). The TiO₂ precursors at 120 °C migrated into the OA as seeds, where nucleation occurs at particles surfaces as shown in Fig. 3. The OA S1 particles size grew from 3 to 5 nm to 6–8 nm. There are a lot of small nanoparticles adhering to the surface of OA particles when the growth temperature was decreased to 80 °C.

The XRD patterns of the OA and OA S1–S3 calcined at 500 °C for 30 min are shown in Fig. 4. For the OA S2 and OA S3, the figure shows that the peaks at 25.2° and 38.5° (TiO₂ anatase, JCPDS No. 21-1272) correspond to the planes (101) and (004) respectively, and confirms the pure anatase phase without any contamination of the other TiO₂ polymorphs. However, the OA S1 reveals the weak peaks of rutile (110) at 2θ = 27.5° (TiO₂ rutile, JCPDS No. 21-1276),

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