



Elucidation of solar cells efficiency by studying the effect of calcination on the synthesized titania nanostructured rods



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ABSTRACT

Elevating calcination conditions of 350 °C, 450 °C, 550 °C, 650 °C and 750 °C on the hydrothermally synthesized titania nanostructured rods were elucidated in terms of morphological change, crystallinity and photovoltaic performance. The field emission scanning electron microscopy (FE-SEM) images shows that the nanomorphology were highly stable and retains its rod structure until the 650 °C, although substantial increase of diameter from 16 nm (350 °C) to 48 nm (650 °C) were observed, ascribed to the sintering influence along with the decrease of surface area from 89.2 m²/g to 19.2 m²/g, respectively. The X-ray diffraction analysis shows a dominant anatase TiO₂ behavior starting from 350 °C to 550 °C with appearance of low intense 33.8°, 43.9° rutile peaks, while on annealing at 650 °C, it transforms to a dominant rutile phase with presence of low intense 25.2°, 48.1° anatase peaks. However, at the 750 °C, the tubular morphology changes to a high crystalline rutile nanoparticles with no trace of anatase phase. Each calcination condition of nanostructured rods tested in dye-sensitized solar cell giving a maximum efficiency of 6.7% at 550 °C. However, the sample at 750 °C shows a low efficiency of 1.5% due to the rutile phase, low surface area, and large grain boundaries resulting in the higher recombination's.

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1. Introduction

Titanium dioxide (TiO₂) is one of the exceptional environmental benign semiconducting materials used in photocatalysis, electrodes for solar cells, sensors and self-cleaning materials owing to its abundance, non-toxicity and biocompatibility [1–5]. Generally, titanium dioxide exists mainly in three different crystalline polymorphs such as anatase (tetragonal, a=b=3.78 Å; c=9.50 Å, band gap=3.23 eV), rutile (tetragonal, a=b=4.58 Å; c=2.95 Å, band gap=3.05 eV) and brookite (rhombohedral, a=5.43 Å; b=9.16 Å; c=5.13 Å, and gap=3.26 eV), all of them consist of a [TiO₆]²⁻ octahedra shared by the edges and corners while keeping and maintaining the overall stoichiometry of TiO₂ [6]. The anatase crystalline form with a tetragonal crystal lattice is most sought and photoactive, hence they are widely and extensively studied among all the titania polymorphs [7]. Depending on the size, shape, crystal lattice structure and surface area the semiconducting properties and band gaps of these titania materials can be tuned, which can strongly affect the photocatalytic and photovoltaic performances, and for that reason low-dimensional nanowires, nanofibres, nanotubes, nanorods were synthesized [8–10]. So far, the nanotubular

1D morphology have received tremendous attention since their larger specific surface area and tubular structure potentially create many reactive sites and efficient electron transfer through the tube channels [11], which leads to a substantial improvement in the photocatalytic properties compared to the bulk materials [12]. To date, several excellent synthetic routes for titania nanotubes/rods have been reported including template synthesis [13], sol-gel synthesis [14], electrophoretic deposition [15], and the hydrothermal synthesis [16]. Among all these techniques, the alkaline hydrothermal method first designed by Kasuga et al. [17] is relatively simple and easy to process. Though by this method final product nanotube/rods possess high surface area but substantially lower in the crystallinity. Which resulting in the high recombinations during the reaction process leading to lower photovoltaic/photocatalytic responses. Furthermore, the as-synthesized 1D nanostructures are in dispute and conflicting reports has been documented such as anatase, hydrogen titanate (H₂Ti₃O₇) [18], ortho-rhombic titanates (Na₂Ti₂O₄(OH)₂) [19], tetratitanates (H₂TiO₄) [20]. Several pioneering effort have been intensively adopted in past decade to modify the post-synthetic low crystallinity of the hydrothermally prepared nanotubes/rods in order to tailor and exploit the excellent optoelectronic properties which includes chemical treatment, annealing effects, removal of the inherent sodium impurities and the ion exchange with the precious metals [21–27].

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Following the first utilization of titania nanoparticles in dye sensitized solar cells (DSCs) as a low cost working electrode material [28], several kind of modified titania based materials are intensively investigated by the manipulation of the morphology, crystallinity and surface characteristics to utilize its uniquely modified properties in the cost effective DSCs. The dye-sensitized solar cells made of two component processes such as light harvesting and charge-carrier transport, where the sensitizer injects electrons in the n-type and holes in the p-type collectors [29]. So, far the dye-sensitized solar cell is dominated by titania nanoparticle and record efficiency of over 12.3% are reported in recent years [30], which can be substantially increased if effectively utilize the novel properties of titania nanotubes/rods/ribbons. Several efforts have been made to use the titania nanotubes/rods/ribbons in dye-sensitized solar cells with an electrochemically grown with a very little success [31,32]. Since, the nanoparticle based DSCs suffers a recombination due to the random orientation of crystals in the films, high diffusion resistant and the involvement of large crystal boundaries likely to occurs [33]. Several methodologies have been devised in order to augment the solar cell efficiencies, among them ID titania nanorods are considered a promising solution since the rod morphology have obvious advantages such as low diffusion resistance, less amount of boundary involvement, efficient charge carrier transport through the rod surface and have high surface areas for the better dye adsorption. The influence of elevating calcination condition in relation to the diameter of the rod morphology, crystallinity and efficiencies are worthy to elucidate. Thus, here in we have studied the influence of temperature influence at 350°, 450°, 550°, 650° and 750°C on the nanorod morphology and then successively applied in the dye-sensitized solar cells in order to get elevating temperature response of the nanorods in DSCs.

2. Experimental

The titania nanorods in this study were prepared by hydrothermal method similar to Kasuga et al. [17] with modification in the silica concentration at the initial nanoparticle synthetic stage with a silica titania ratio of 10:90, as it has been previously reported [34]. Then these nanoparticles were used for nanorod synthesis at higher temperature of 190 °C for 24 h in the Teflon lined autoclave with 10 M aqueous NaOH solution, which possess a rod diameter of ~13 nm. Such prepared low intense crystalline titania nanorods were annealed at 350 °C, 450 °C, 550 °C, 650 °C and 750 °C in ambient for 2 h and then studied to elucidate the nanomorphology, crystal phase and efficiencies. For the dye-sensitized solar cell (DSC) device fabrication, firstly a FTO glass (TEC-8, Pilkington) was cut in 1.5 cm x 1.5 cm and cleaned successively by water, ethanol and acetone for 15 minutes each in ultrasonic bath, respectively. A doctor blade method is applied to make the films from the annealed (350°, 450°, 550°, 650 °C) titania nanostructured rods (0.4 g) by making slurry with the addition of polyethylene glycol (Junsei, MW 20,000) and ethanol (2 ml). The coated substrate was then transferred to the electrical muffle furnace and annealed for 30 min at 450 °C, successively other sample's also done in the same way. The dye solution was made by using N719 at 0.3 mM concentration with acetonitrile: tert-butanol = 50:50 vol% with 0.075 mM, then the working electrode was dipped in it in dark at room temperature for 24 h. The transparent counter electrode of FTO was prepared and cleaned in same manner and was dipped in chemically deposited 10 mM hydrogen hexachloroplatinate (IV) hydrate (99.9%, Aldrich) in 2-propanol. The working electrode and the counter electrode were sandwiched together by using a 60 μm thick Surlyn (Solarinox) spacer at 90 °C for 10 s at some pressure. Then the prepared electrolyte was injected through the hole by using vacuum injection method then again covered with surlyn spacer to block the hole, the electrolyte was prepared as 0.5 M

1-Hexyl-2,3-dimethylimidazolium iodide (HDMII, 99.9%, C-tri), 0.02 M Iodine (Aldrich), 0.5 M 4-tert-butylpyridine (TBP, Aldrich), 0.05 M Lithium Iodide (Aldrich). The photo-electrochemical properties of the solar cell were recorded by the current-voltage characteristics (digital source meter; model 2400, Keithly) in illumination (AM1.5, 100 mW/cm²; model ORIEL-Sol-3A, Newport). The active area of the each cell in this study were ~ 0.25 cm². The synthesized product were characterized by FE-SEM images taken from field emission scanning electron microscope (TESCAN MIRA LMH2, operated at 15 kV) and Transmission electron microscopy by (TEM, JEM- 2100). XRD diffraction pattern was analyzed by operating at 30 kV and 40 mA by using Cu K α radiation with scanning of 4/min (Rigaku, D/Max-2500). UV-Vis spectra was recorded by UV spectrophotometer (Shimadzu UV 1201). The surface area was determined by using the Brunauer-Emmett-Teller (BET) equation using N₂ adsorption/desorption carried out at 77.3 K by ASAP 2020 X tended Pressure Sorption Analyzer.

3. Result and discussion

Fig. 1 (a–e) shows the field emission scanning electron microscope (FE-SEM) images of the annealed nanostructured rods at 350 °C, 450 °C, 550 °C, 650 °C and 750 °C in the ambient, respectively. It was clearly observed that with successive calcinations (350 °C to 650 °C) the rod diameter changes substantially. When the nanorods calcined at 350 °C (Fig. 1a) a diameter of 16 nm with an average length of more than 800 nm were observed, and when they were further heated to 450 °C (Fig. 1(b)) the diameter increases to 21 nm. At this stage it is interesting and worthy to observe that the nanorods were individually segregated and were relatively shorter in the length as compared to the 350 °C sample. While those nanorods that calcined at 550 °C and 650 °C (Fig. 1 (c,d)), the diameter successively increases to 33 nm and 48 nm, respectively. The reduced in the aspect ratio were observed where a bunch of nanorods joined together/coalesces in close proximity and loss of the individuality clearly seen, possibly due to sintering effect which is more pronounced at this stage due to higher calcination conditions. It is worthy to observe that, at higher temperature (650 °C, Fig. 1(d)) some breakage of the thick nanorods started appearing probably due to the contraction of interlayer distances caused by internal strain incorporated in the rod wall by thermal energy, however, at the calcination to 750 °C (Fig. 1(e)) it rendered all of the nanorods in the poly size nanoparticle. Fig. 2(a,b) shows the transmission electron microscopy (TEM) image of the calcined nanorods at 350 °C and 550 °C, respectively. It can be seen that the calcined nanorods where the diameter shows, 16 nm and 32 nm are consistent with the SEM images. It can be also found that the 350 °C calcined sample possess a corrugated structure at the sideways of individual nanorods which is not observed in 550 °C sample, possibly the influence of sintering. The selected area electron diffraction pattern (SAED) of the sample calcined at 550 °C is shown in the Fig. 3(c) where a clear ring pattern is observed from the crystal phases of (101), (004), (200) are well consistent with the crystallinity of the analyzed XRD patterns.

Fig. 3. depicts X-ray diffraction measurement to analyze the crystal phases of the annealed nanostructured rods over a 2 θ range of 20–70° with a step size of 4°/min at 350 °C, 450 °C, 550 °C, 650 °C and 750 °C, respectively. It can be seen from the figure that the samples calcined at 350 °C to 550 °C, the dominant peak position at 2 θ located at 25.2°, 37.4°, 48.0°, 54.2°, 55.2°, 62.2°, 67.1° corresponding to the anatase crystal phases of (101), (004), (200), (105 and 211), (204), (220) which are consistent with the (JCPDS card no. 21–1272). However few rutile peaks also emerged in the 450 °C calcined sample although they were not clear in 350 °C calcined sample at the peak locations of 33.8°, 43.9° which

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