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Energy Procedia 34 (2013) 555 – 562

Energy Procedia

10th Eco-Energy and Materials Science and Engineering

(EMSES2012)

# Characterization of Flower-like Titanate and Titania Nanowires on Titanium Plate Substrate

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**Abstract**

Flower-like structure titanate and titania on titanium plate substrate composed of nanowires were synthesized by hydrothermal method at 150 °C for 2 h using titanium plate substrate as the starting material. The crystalline structure, shape and size of the prepared sample were characterized by X-ray diffraction (XRD) and scanning electron microscope (SEM). SEM images showed flower-like structure with uniform size about 2 μm. The flower-like structure composed of nanowires with diameter about 20-50 nm. Titanate and titania could be controlled by washing process and calcination temperature. This synthesis method provides a simple route to fabricate nanostructured titanate and titania on titanium plate substrate for dye-sensitized solar cell and photocatalytic activity applications.


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*Keyword:* Nanowires, Titanium, Titanium dioxide, Hydrothermal

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doi:10.1016/j.egypro.2013.06.785

## 1. Introduction

Titanium (Ti) has superior corrosion resistance due to the existence of passive film which consisted of $TiO_2$. It is expected that the application of a metal sheet as a support of $TiO_2$ in dye-sensitized solar cells cannot only reduce the cost of solar cell, but can also contribute to improve the performance of the solar cells by reducing internal resistance [1]. The synthesis and characterization of one-dimensional (1-D) nanostructured (nanotubes, nanorods, and nanowires) have received considerable attention due to their unique properties and novel application [2]. Hydrothermal synthesis has become one of the most important and promising new material fabrication method for nanoscale materials and nanotechnology [3–12]. In our previous works, titanate, $TiO_2$ (B), and anatase $TiO_2$ nanofibers were synthesized by hydrothermal method using natural rutile sand as the starting material to reduce the production cost, however, the diameter of the nanofibers were about 20-100 nm [4–5].

In this study, flower-like structure titanate and titania on titanium plate substrate composed of nanowires were synthesized by hydrothermal method at 150 °C for 2 h using titanium plate substrate as the starting material.

## 2. Experimental

In a typical synthesis, Ti plate substrate (Showa Co. Ltd.) was washed with acetone and then put into a Teflon-lined stainless steel autoclave that containing 50 ml of 10M NaOH aqueous solution and 2 ml of $H_2O_2$. The autoclave was heated at 150 °C for 2 h. After the autoclave was naturally cooled to room temperature, the obtained product was washed with HCl aqueous solution, distilled water for several times, followed by drying at 100 °C for 12 h. The crystalline structure of samples was evaluated by X-ray diffraction (M18X-CE, Bruker AXS, Japan, Cu-Ka radiation, 40kV and 100mA). The microstructure of the prepared products was analyzed by scanning electron microscopy (SEM, JEOL JSM-6500FE) (Fig. 1).

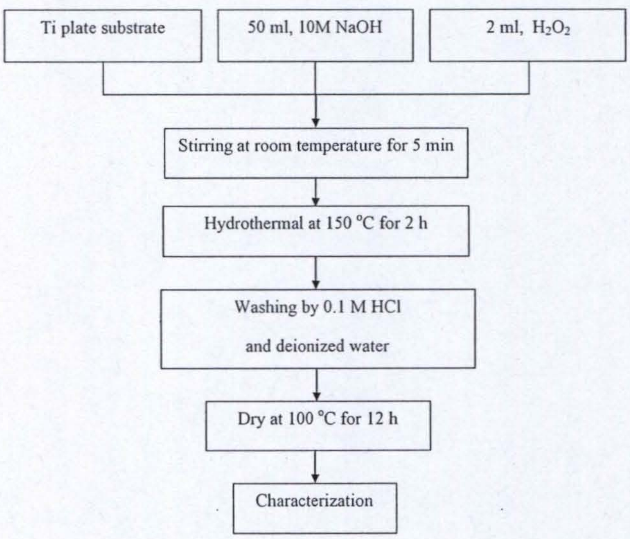


Fig. 1. Schematic representation of the preparation of flower-like titanate.

## 3. Results and Discussion

The XRD patterns of the Ti plate substrate, the as-synthesized sample, and the sample calcined at 500 °C for 2 h are shown in Fig. 2 (a-c)). The structure of the as-synthesized sample is similar to that expected for a layered titanate $H_2Ti_xO_{2x+1}$, probably trititanate ($H_2Ti_3O_7$) [4–5]. No diffraction peaks of other impurities (such as NaCl) were observed. This result is due to the Na content in the samples was reduced by HCl and water treatment [12].

The sample calcined at 500 °C for 2 h consisted of anatase $TiO_2$ and small peak of $TiO_2$ (B) structure. The peaks were rather sharp, which indicated that the calcined sample $TiO_2$ had relatively high crystallinity. Titanate and titania could be controlled by washing process and calcination temperature [5].

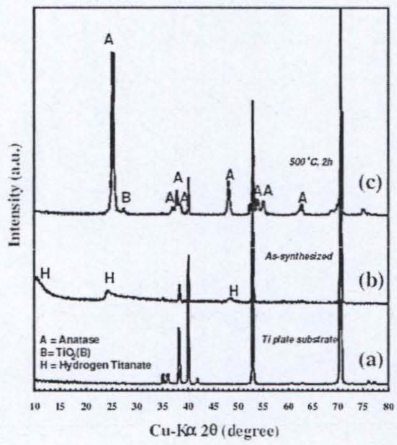


Fig. 2. XRD patterns of (a) Ti substrate, (b) the as-synthesized sample, and (c) the sample calcined at 500 °C for 2 h.

The SEM images showed flower-like structure with uniform size about 2 μm (Fig.3 (a-c)). The flower-like structure composed of nanowires with diameter about 20-50 nm. Furthermore, the literature reported the flower-like structure showed high specific surface areas leading to high photocatalytic activity due to large contact area [13–14]. After calcination at 500 °C for 2 h, the structure changed to nanowires/nanoparticles composite (Fig.4 (a-c)). In order to confirm nanowires (solid structure) or nanotubes (hollow structure). Normally, the $TiO_2$-derived nanotubes after heat-treatment at 400 °C (anatase phase) were destroyed then change into particles. This reason limited to utilize at high temperature (typically at 500-800 °C). To avoid this problem, nanowires or nanofibers were initially alternative due to their maintain wire- or fiber-like structure at high temperature [12, 15–18]. The formation mechanism of flowerlike $TiO_2$ is proposed to proceed via a three-step process as depicted in Fig. 5. From the SEM research (Fig. 4), it can be concluded that flowerlike $TiO_2$ nanostructures can be obtained via a nucleation–dissolution–recrystallization growth process [19–20].

Recently, Wang et al [21] reported the flower-like anatase $TiO_2$ microarray exhibited much stronger absorption in the range from 320 to 800 nm and a remarkable red shift in the band gap transition, while absorption spectra of P-25 appeared at wavelength shorter than 400 nm. Interesting, the flower-like anatase $TiO_2$ showed the photocatalytic activity of degradation of methyl orange (MO) under UV irradiated than that of the P-25 [21]. The unique morphology and hierarchical structure of flower-like anatase $TiO_2$ may play an important role in enhancing the UV photocatalytic activity of $TiO_2$ due to effective light-scattering inside the gaps between nano-flakes, resulting in the photogenerated electrons and holes to participating effectively in the photocatalytic degradation of contaminants [21–22].

This results, indicated that their characterization reveals a special nanostructured material which not only determines the ultimate structure but also provides a unique material with potential applications for electronic, photonic and biology such as dye-sensitized solar cell [1, 23], lithium-ion batteries [24], sensor [25], biomedical [26] and photocatalytic activity [27–28].

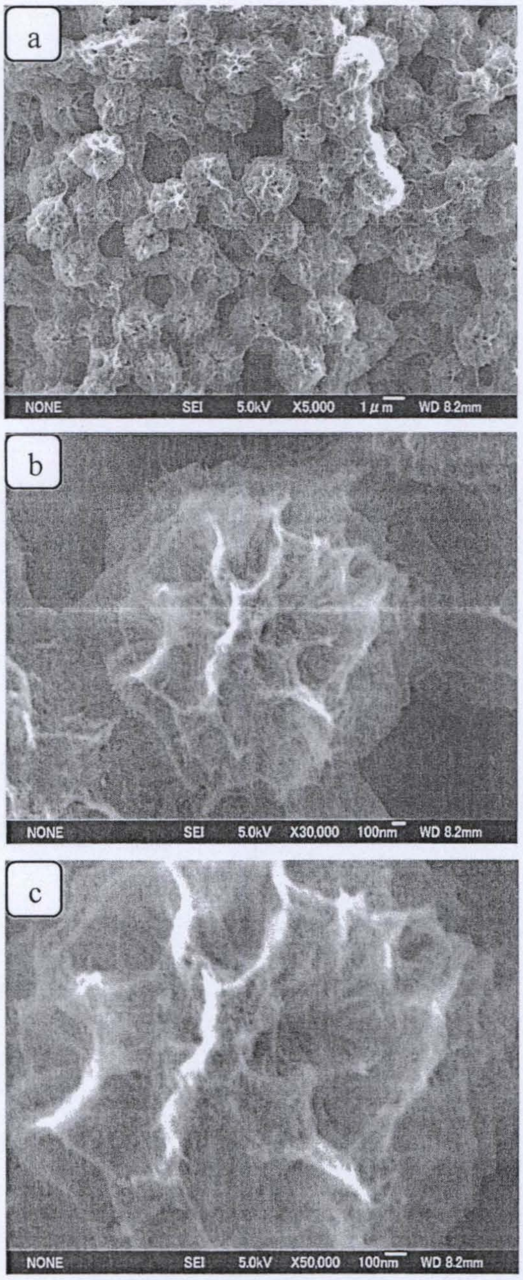


Fig. 3. SEM images of flower-like nanowires titanate (as-synthesized) on Ti plate substrate at (a) x 5,000, (b) x 30,000 and (c) x 50,000 magnified

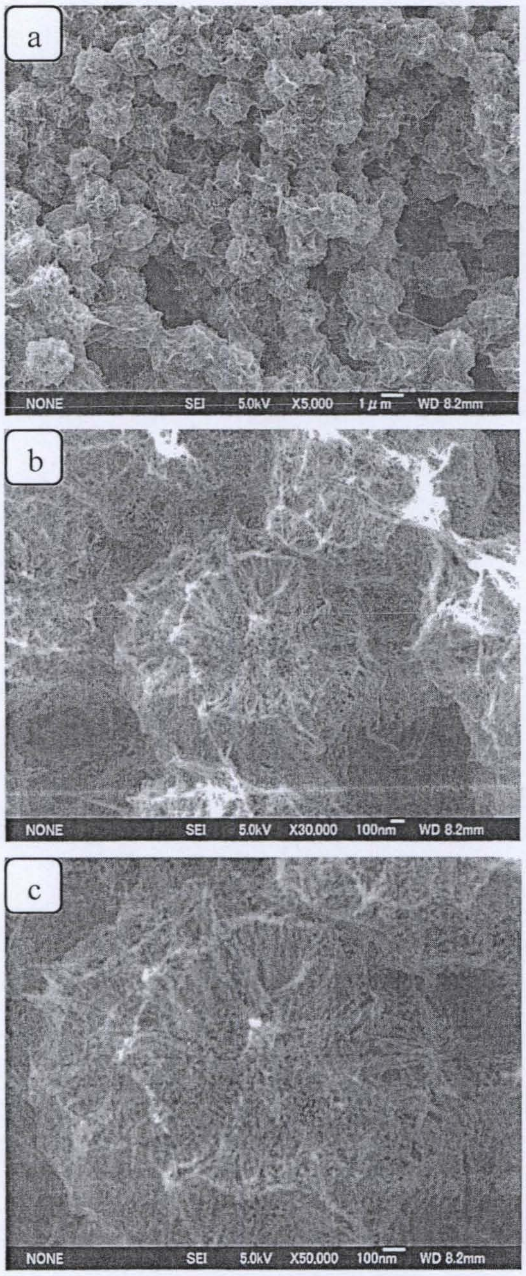


Fig. 4. SEM images of flower-like nanowires titanate (calcined at 500 °C for 2 h) on Ti plate substrate at (a) x 5,000, (b) x 30,000 and (c) x 50,000 magnified.

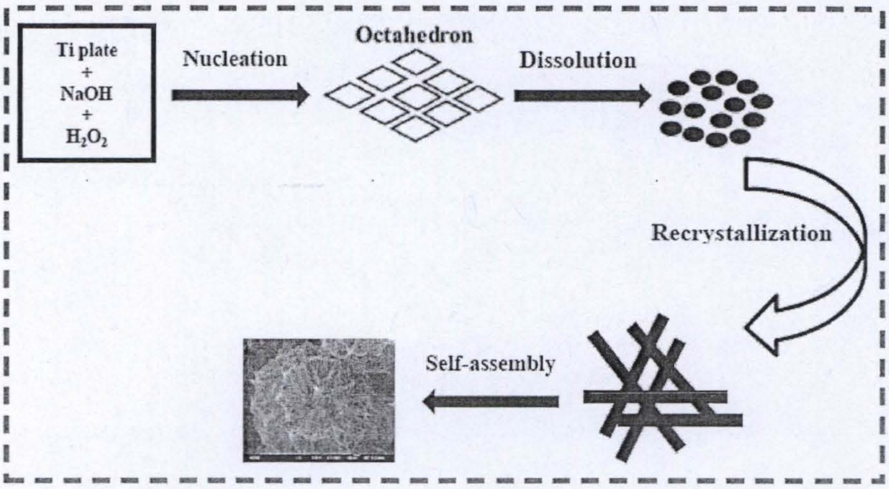


Fig. 5. Schematic illustration of the formation and shape evolution of flowerlike $TiO_2$ nanostructures in the hydrothermal process.

## 4. Conclusion

Flower-like structure titanate and titania on titanium plate substrate composed of nanowires were synthesized by hydrothermal method at 150 °C for 2 h using titanium plate substrate as the starting material. SEM images showed flower-like structure with uniform size about 2 μm. The flower-like structure composed of nanowires with diameter about 20-50 nm. The as-synthesized flower-like structures were $H_2Ti_3O_7$ crystal phase. The prepared sample calcined at 500 for 2 h consisted of anatase $TiO_2$ and small peak of $TiO_2$ (B) phase. This synthesis method provides a simple route to fabricate nanostructured titanate and titania on titanium plate substrate for dye-sensitized solar cell, lithium-ion batteries, sensor, biomedical and photocatalytic activity applications.

## Acknowledgements

This work was supported by National Research Council of Thailand (NRCT). The authors would like to express gratitude to Showa Co. Ltd. for supporting the Titanium plate substrate, the College of Nanotechnology, King Mongkut's Institute of Technology Ladkrabang (KMITL) and the Nanotechnology for Textile and Polymer Research Group (NanoTeP) of the Faculty of Engineering, Rajamangala University of Technology Thanyaburi (RMUTT), Thailand.

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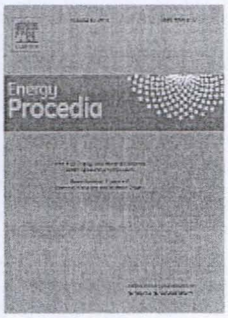


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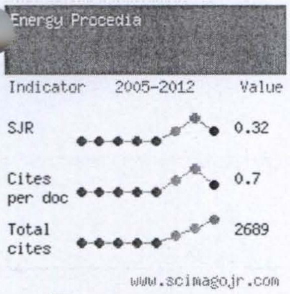


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