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Studies on transformation of titanate nanotubes into nanoribbons

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ABSTRACT

High aspect ratio titanate nanostructures were synthesized by simple hydrothermal treatment and the nature of two distinct morphologies, hollow nanotubes and titanate nanoribbons was explored as a function of hydrothermal processing conditions. The samples were characterized by means of SEM, XRD and TEM. The specific surface area of the final products was determined by Brunauer-Emmett-Teller (BET) method. It has been found that hydrothermal temperature and the treatment duration have a strong effect on the morphological control of the resulting products. Transformation of nanotubes into nanoribbons was observed with increase in the treatment temperature from 180 °C to 200 °C which became more dense with further increase in the temperature from 200 °C to 220 °C and treatment duration from 12 h to 24 h.

1. Introduction

In recent years, fabrication of nanomaterials with a controllable size and shape has been of great scientific and technological interest due to their unique properties and potential applications. Significant efforts have been taken in the synthesis of various 1D nanoscale materials over the past few years. Among a variety of materials high aspect ratio titanate nanostructures have been of special interest due to titanium dioxide's strong resistance to chemical and photocorrosion, its safety and low cost and biological harmlessness. They have remarkable applications in the field of photocatalysis, dye sensitized solar cells, gas sensors, electrodes for Li battery and so on [1–4].

On the basis of the pioneering work of Kasuga et al. research efforts on titanates were at first concentrated on structure elucidation of titanate nanotubes [5]. TiO_2 nanotubes have been synthesized by various techniques correlated to high and low temperature techniques such as chemical vapor deposition, thermal evaporation and hydrothermal method [6]. However substantial research studies have indicated that hydrothermal route is a powerful and promising strategy for preparing 1D nanomaterials because of its advantages such as simple procedure and low cost [7–14]. It has been claimed that this process yields single crystal nanotubes with a uniform outer diameter of around 10 nm and a length of several 100 nanometers [15–17].

Although till date, most researchers have focused on the formation of these nanotubes, now a days, nonhollow titania nano objects are also being investigated by several groups [18–21]. However statements in the literature concerning the impact of the various preparation conditions on the resulting structure are often conflicting and inconsistent. It was reported by Weng et al. that increasing the hydrothermal duration leads to the increase in nanotubes length until there is no further increase in the length if the treatment duration exceeds 24 h [22]. Yuan et al. reported that the optimum yield of nanotube formation is achieved at 150 °C, whereas the formation of high aspect ratio nanoribbons is observed only at temperature higher than 180 °C [23]. Recently Elsanuosi et al. synthesized titanate nanotubes and nanoribbons by hydrothermal treatment and found that hydrothermal temperature and treatment duration had a strong effect on the morphology control of the resulting products. They have suggested that more studies on the effect of hydrothermal conditions should be performed [24].

Taking all these factors into consideration, the present study has been focused on the investigations of the relationship between hydrothermal conditions, size and morphology of the high aspect ratio products.

2. Experimental details

2.1. Sample preparation

TiO_2 nanotubes/nanoribbons were prepared using a chemical process similar to that described by Kasuga et al. [5]. In a typical preparation procedure, 1 g of TiO_2 nanopowder (average size 32 nm) was added to 100 mL of 10 M NaOH and stirred for 30 min in a beaker. The mixture was then transferred into a teflon-lined stainless steel autoclave of 100 mL capacity till about 80% of its total volume, aging the suspension at different temperatures (180 °C, 200 °C and 220 °C) and time durations (12 h and 24 h). After the autoclave was naturally cooled to room temperature, the obtained sample was subsequently filtered and washed with distilled water to get its pH neutral. Then acid washing was done with 0.1 M HCl for 6 h. Subsequently the solution was washed

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several times until the pH value of the solution reached to 7, and then the sample was dried at 70 °C for 6 h.

2.2. Phase structure and morphological characterization

The phase structure, morphology and composition of the as-prepared titainia nanotubes/nanoribbons were examined by using various techniques like scanning electron microscopy (SEM, Philips FEI-XL30) operating at 25 kV and by X-ray powder diffraction (XRD, Panlytical XpertPRO diffractometer with CuKα radiation, $\lambda = 0.1542$ nm, 40 kV, 40 mA). The specific surface area of the as-prepared nanostructure was determined by Brunauer-Emmett-Teller (BET) method by using nitrogen adsorption data at 473 K on a Micromeritics Analyzer ASAP 2020.

3. Results and discussion

3.1. SEM analysis

Fig. 1 shows the SEM images of the samples treated at 180 °C, 200 °C and 220 °C for different hydrothermal durations. Fig. 1a shows that the morphology of the products obtained by treating the sample at 180 °C for 12 h was anomalistic. After 24 h hydrothermal treatment duration, formation of some one dimensional structures could be seen (Fig. 1b). Increasing the treatment temperature to 200 °C revealed that at 12 h, some nanotubes were formed with diameters of about 10 nm and lengths up to several hundreds of nanometers (Fig. 1c). The formation of few nanoribbons was also observed beside the nanotubes, which became more dense with increase in treatment duration from 12 h to 24 h (Fig. 1c-d). The nanoribbons had widths ranging from 50 to 200 nm and lengths of several micrometers. At higher temperature of 220 °C (Fig. 1e-f), SEM observations showed that dense nanoribbons had already been formed at 12 h. At longer treatment duration of 24 h, bundles of very long and wide nanoribbons were noticed having lengths of several tens of micrometers and width variations between 50 nm and 500 nm.

It is clear from these results that the hydrothermal treatment duration and treatment temperature has a strong effect on the morphological features of the resulting products. We can see that with increase in the treatment duration, more dense pure nanoribbons would be formed. The temperature has the similar effect i.e. at higher temperature, dense nanoribbons are formed in shorter duration.

3.2. XRD analysis

To observe the crystallographic changes of the sample due to the change in the morphology from nanotubes to naoribbons, analysis with XRD was made to all products and Fig. 2a-g shows the patterns obtained.

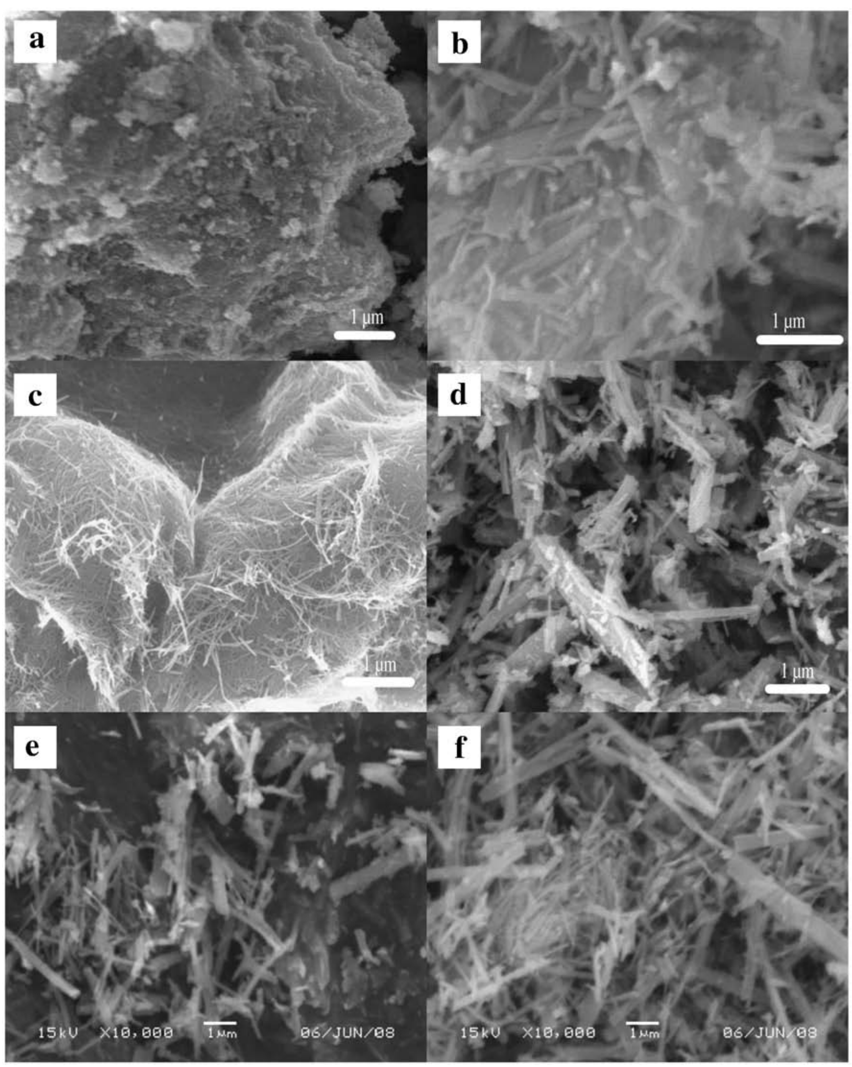

Fig. 1. SEM images of the samples treated at different temperatures and different durations (a) 180 °C 12 h; (b) 180 °C 24 h; (c) 200 °C 12 h; (d) 200 °C 24 h; (e) 220 °C 12 h; (f) 220 °C 24 h.