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Photocatalyst Nanofibers Obtained by Calcination of Organic-Inorganic Hybrids

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1. Introduction

Electrospinning (ES) is one of the most useful techniques to form nanofibers in a diameter of several hundred nanometers (Doshi & Reneker, 1995, Buchko et al., 1999, Huang et al., 2003). The diameter of the nanofibers produced by ES is at least one or two orders of magnitude smaller than those of conventional fiber production methods like melt or solution spinning. As a result, the electrospun nanofibers have high specific surface area (Yamashita, 2007). These nanofibers are well-suited to be used as chemical reaction fields (Nakane et al., 2005, 2007).

Much attention has been paid to the formation of both organic polymeric nanofibers and inorganic nanofibers using ES (Ramakrishna et al., 2005). Many kinds of inorganic nanofibers (SiO_2 , Al_2O_3 , ZrO_2 , NiCo_2O_4 , and so on) have been obtained by calcination of organic-inorganic hybrid precursor nanofibers formed by ES (Guan et al., 2004, Shao et al., 2004, Chronakis, 2005, Panda & Ramakrishna, 2007, Krissanasaeranee et al., 2008). The formation of TiO_2 nanofibers have been also reported by several research groups. Li and Xia formed anatase-type titanium oxide (TiO_2) nanofibers by the calcination of poly(vinyl pyrrolidone) (PVP)-Ti tetraisopropoxide (TTIP) hybrid nanofibers at 500°C in air (Li & Xia, 2003). The TiO_2 nanofibers obtained would be a useful material for a photocatalytic reaction, but their usage has not been investigated. Ethanol has been used as the solvent of the spinning solution to form the hybrid precursor nanofibers. Therefore, a spinneret could be stopped up by a solid material because ethanol will evaporate from the tip of the spinneret during the spinning. Furthermore, TTIP is very easily hydrolyzed, and thus a water-free condition is required for the use of TTIP on ES. Another groups also formed TiO_2 nanofibers by calcination of TiO_2 -PVP and TiO_2 -poly(vinyl acetate) precursors which were formed by ES using organic solvents such as ethanol and dimethylformamide (Kim et al., 2006, Nuansing et al., 2006, Kumar et al., 2007, Ding et al., 2008).

Li and Xia reported the formation of TiO_2 hollow-nanofibers (nanotubes) by ES of two immiscible liquids (TTIP-PVP ethanol solution and heavy mineral oil) through a coaxial, two-capillary spinneret, followed by selective removal of the cores and calcination in air (Li & Xia, 2004). The TiO_2 nanotubes with uniform and circular cross-sections were obtained by the method. Kobayashi et al. reported the preparation of TiO_2 nanotubes using the gelation (self-assembly with a rodlike fibrous structure) of an organogelator (It is not ES.) (Kobayashi et al., 2000, 2002). The organogelator is a cyclohexane derivative that was specially

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synthesized by this research group. The outer and inner diameters of the TiO₂ nanotubes obtained were 150-600 nm and 50-300 nm, respectively. We considered that the TiO₂ nanotubes would be easily formed by using the ES process without organogelators. In this study, we formed TiO₂ nanofibers (Nakane et al., 2007) and TiO₂ nanotubes (Nakane et al., 2007) by calcination of new precursor nanofibers of poly(vinyl alcohol) (PVA)-titanium compound hybrids. The precursor nanofibers were formed by using ES with water as a solvent. It is most safety method to use water as a solvent on ES. The photocatalytic reaction using the TiO₂ nanofibers obtained was also investigated.

2. Materials and methods

2.1 Formation of PVA-Ti compound hybrid precursor nanofibers and TiO₂ nanofibers

Two types of precursor nanofibers were formed using ES.

Precursor-1: PVA (degree of polymerization: 1500) 10 wt% aqueous solution was prepared. Titanium lactate (TL) [(OH)₂Ti(C₃H₅O₂)₂] (5 g) was added to the PVA solution (10 g) to produce transparent PVA-TL mixed solution (spinning solution). TL was kindly gifted from Matsumoto Chemical Industry Co., Ltd., Japan (TC-310, content: TL 35-45%, 2-propanol 40-50%, water 10-20%). The mixed solution was loaded into a plastic syringe (2 ml) equipped with a needle. The solution extrusion rate was 0.015 ml/min. A voltage of 25 kV was applied to the needle, and the PVA-TL hybrid nanofibers were then deposited on a collector. The collector (copper plate) was grounded, and the distance between the tip of the needle and the collector was 10 cm. The PVA-TL hybrid nanofibers obtained were used as a precursor of TiO₂ nanofibers.

Precursor-2: Pure PVA nanofibers formed by ES were immersed in a titanium alkoxide [titanium tetraisopropoxide (TTIP)] (10wt%)-ethanol solution for 10 minutes. The treated nanofibers were washed in fresh ethanol and then PVA-TTIP hybrid precursor nanofibers were obtained.

These precursor nanofibers obtained were calcined up to a given temperature in an electric furnace (in air), and TiO₂ nanofibers were formed.

2.2 Apparatus and procedure

The structure of the nanofibers was observed by scanning electron microscope (SEM) (Hitachi S-2400, Japan). Thermogravimetric (TG) analysis was performed in air and a heating rate of 10 °C/min (Shimadzu DTG-60, Japan). X-ray diffraction (XRD) measurement was taken using a CuK α with a Ni filter (40 kV, 30 mA) (Shimadzu XRD-6100, Japan). The nitrogen adsorption isotherms (-196°C) of the TiO₂ nanofibers were measured by Micromeritics TriStar 3000, USA.

The photocatalysis of the TiO₂ nanofibers was evaluated using the photocatalytic decomposition of methylene blue (3,7-bis(dimethylamino) phenothiazin-5-ium chloride; C₁₆H₁₈ClN₃S) (Nakane et al., 2007). The TiO₂ nanofibers formed (5 mg) were dispersed in methylene blue (1 \times 10⁻⁵ mol/l) aqueous solution (50 ml). Three ml of test liquid was taken from this solution and fed in a quartz cell. The test solution was irradiated with white light using an extra-high pressure mercury vapor lamp (Ushio Inc., Japan), and the absorbance at 665 nm, which is the maximum absorption wavelength of methylene blue, was measured by an absorptiometer (JASCO, CT-109, Japan). The decomposition rate of methylene blue was calculated from the absorbance.

3. Results and discussion

3.1 TiO₂ nanofibers obtained from PVA-TL hybrid nanofibers

Fig. 1 shows the SEM image of PVA-TL hybrid nanofibers (precursor-1). The fiber diameters of the PVA-TL nanofibers are 200-350 nm, and the fibers have a smooth surface without macropores. The specific surface area and the pore volume of the hybrid nanofiber are 1.80 m²/g and 0.00764 cm³/g, respectively. Thus, the hybrid nanofiber is considered a nonporous material.

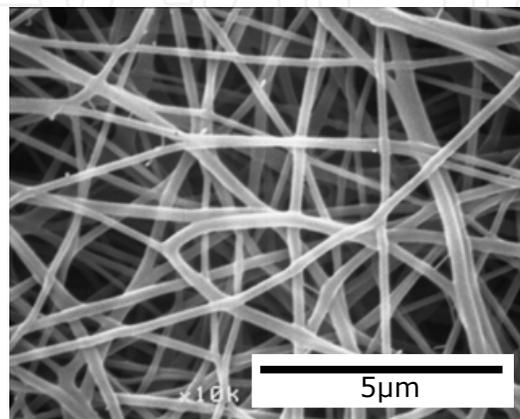


Fig. 1. SEM image of PVA-TL hybrid nanofibers (precursor-1).

Fig. 2 shows TG curves of the pure PVA and the PVA-TL hybrid nanofibers. The weight residue of pure PVA becomes zero at 550°C, and that of the PVA-TL hybrid is 25% at 600°C. White residues (TiO₂) were obtained after measurement for PVA-TL hybrid.

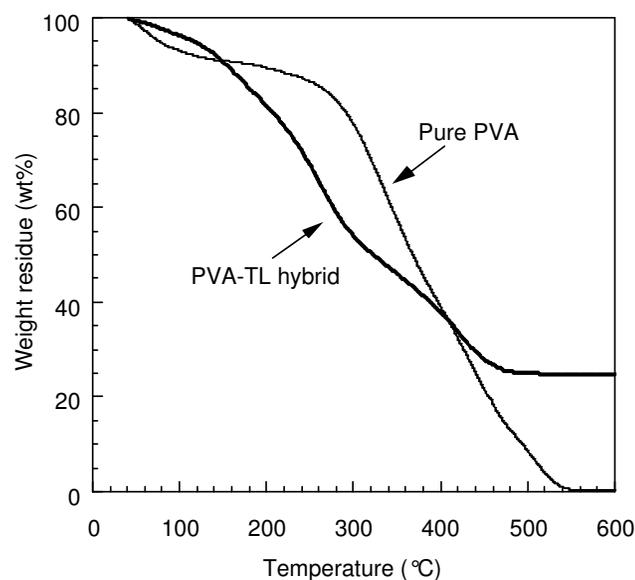


Fig. 2. TG curves of pure PVA and PVA-TL hybrid nanofibers.

Fig. 3 shows the SEM image of the TiO₂ nanofibers obtained by the calcination of PVA-TL hybrid nanofibers at 400°C for 5 hours. Compared to the image shown in Fig.1, the fiber diameter of the TiO₂ nanofibers is 70-80% of that of the PVA-TL hybrid nanofibers, with the

space between the fibers made denser by calcination. The residues were brittle, but maintained the shape of the PVA-TL hybrid non-woven mat, although shrinkage occurred due to the calcination.

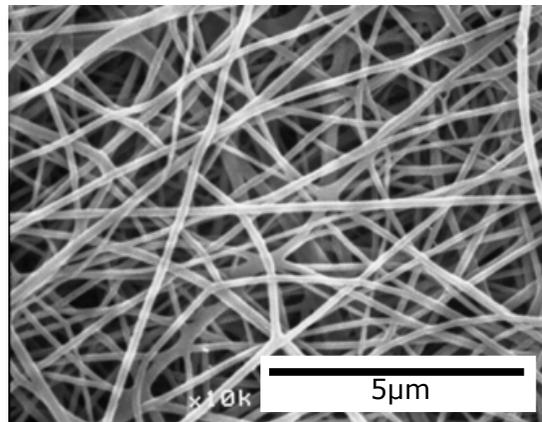


Fig. 3. SEM image of TiO_2 nanofibers obtained by calcination at 400°C for 5 hours in air.

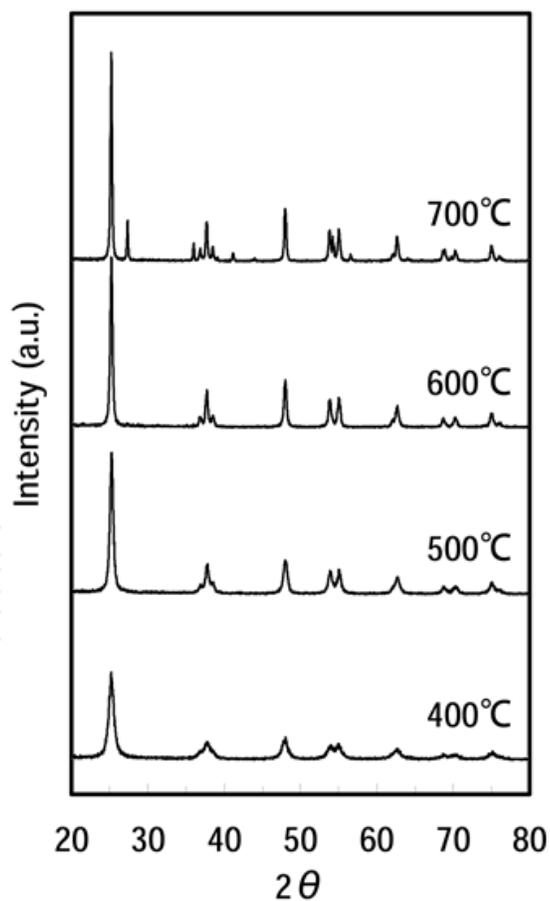


Fig. 4. XRD curves of TiO_2 nanofibers calcined at prescribed temperatures for 5 hours in air.

Fig. 4 shows XRD curves of the TiO_2 obtained by calcination of the PVA-TL nanofibers at $400\text{--}700^\circ\text{C}$ for 5 hours. Anatase-type TiO_2 is formed at $400\text{--}600^\circ\text{C}$, and the peak intensities

increase with calcination temperature. Rutile-type (rutile-anatase mixed) TiO_2 is formed at 700°C . It is well-known that anatase is superior to rutile for photocatalysis. Thus, a calcination temperature of $600\text{--}700^\circ\text{C}$ would be an effective condition when using the TiO_2 nanofibers as a photocatalyst.

Fig.5 shows the relationship of the calcination temperature of the hybrid nanofibers and the pore characteristics (specific surface area and pore volume) obtained from the nitrogen adsorption isothermes (-196°C) of the TiO_2 nanofibers. The specific surface area and pore volume of the TiO_2 nanofibers decreases with increasing the calcination temperature. This is due to the sintering of the TiO_2 by the calcination. The average pore diameters (d_p), which were assumed to be cylindrical in shape, were based on the specific surface area (S) and pore volume (V) for each TiO_2 nanofiber: $d_p=4V/S$. The d_p of the TiO_2 nanotubes were 3.8 nm (calcination temperature: 400°C), 7.4 nm (500°C), 17.5 nm (600°C), and 44.0 nm (700°C). Consequently, the TiO_2 nanofibers obtained in this study are classified as mesoporous materials.

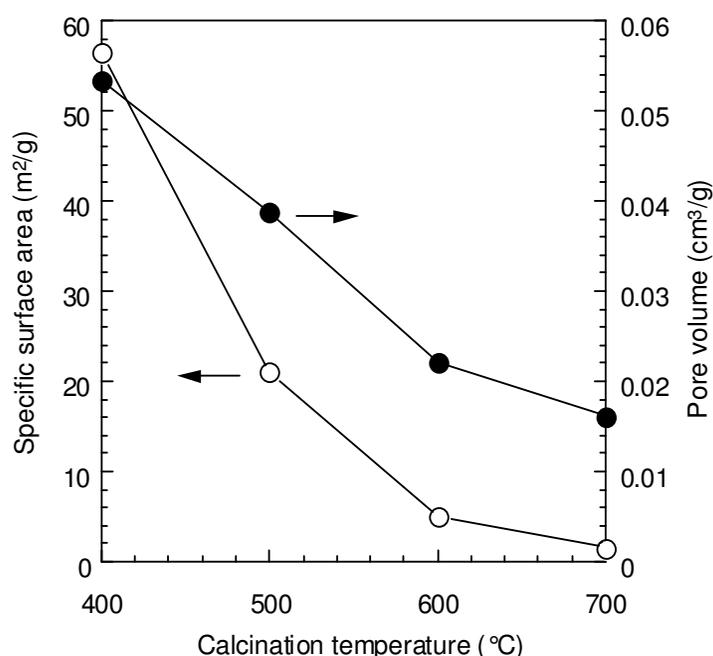


Fig. 5. Effect of calcination temperature upon the pore characteristics of the TiO_2 nanofibers.

3.2 TiO_2 nanotubes obtained from PVA-TTIP hybrid nanofibers

Fig.6 shows the SEM images of (a) pure PVA nanofibers formed by ES, and (b) PVA-TTIP hybrid precursor nanofibers (precursor-2). The fiber diameter of a PVA nanofiber is *ca.* 200 nm, and the fiber diameter of the precursor nanofiber becomes 1.3 times larger than that of the PVA nanofiber. Both fibers have a smooth surface. White residues (TiO_2) were obtained by calcination of the hybrid precursor nanofibers at 500°C for 5 hours. The sample weight after calcination was 12.1% of the one before calcination.

Fig.7 shows the SEM image of the residue after calcination of the precursor nanofiber at 500°C for 5 hours. As can be seen from this image, hollow TiO_2 nanofibers (TiO_2 nanotubes: outer diameter, *ca.* 440 nm, inner diameter, *ca.* 270 nm) were formed. The nanotubes obtained are seemed to have a uniform diameter as compared to the diameter range reported by Kobayashi et al. (Kobayashi et al., 2000). We determined that a precursor nanofiber with a

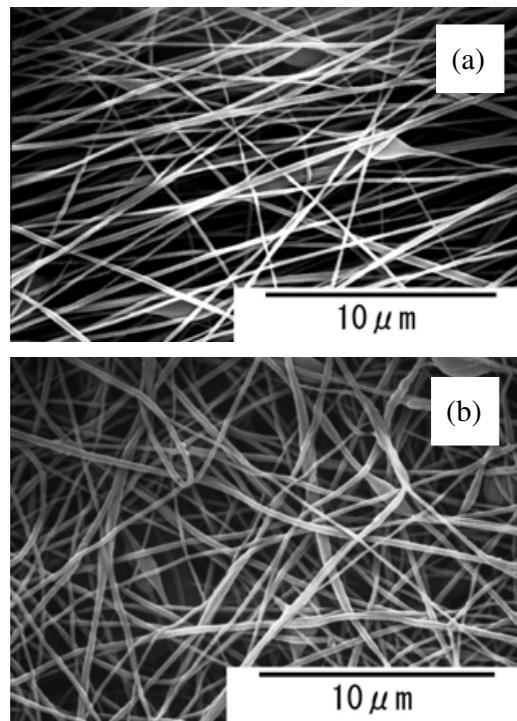


Fig. 6. SEM images of (a) pure PVA nanofibers and (b) PVA-TTIP hybrid nanofibers (precursor-2).

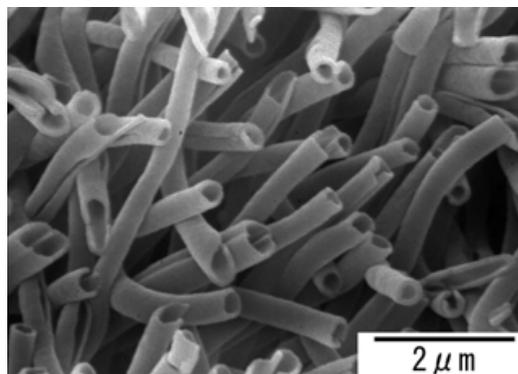


Fig. 7. SEM image of TiO_2 nanotubes formed by calcination of PVA-TTIP hybrid nanofibers.

skin-core structure would be obtained by our method; the skin layer is a PVA-TTIP hybrid, and the core is pure PVA. TTIP will penetrate into the PVA matrix when the PVA nanofibers are immersed in TTIP-ethanol solution, and a PVA-TTIP hybrid layer will be formed. The interaction between the PVA and TTIP could not be identified, but the hybrid would be formed by a coordination bond between the titanium and the oxygen of the hydroxyl group on the PVA molecules (Nakane et al., 2003). The structure of the TiO_2 nanotubes obtained would be reflected in the skin-core structure of the precursor nanofibers. Schematic illustration of the formation of TiO_2 nanotubes by our method is shown in Fig.8.

The diameter of the nanotube can be controlled by changing the diameter of the pure PVA nanofiber. However, nanotubes were not obtained when the outer diameter was *ca.*200 nm (Fig.9 (a), formed of non-hollow nanofibers) or *ca.*700 nm (Fig.9 (b), breaking of TiO_2 tube wall). Thus, the range of the outer diameter which can be formed in this study is 300–600 nm.

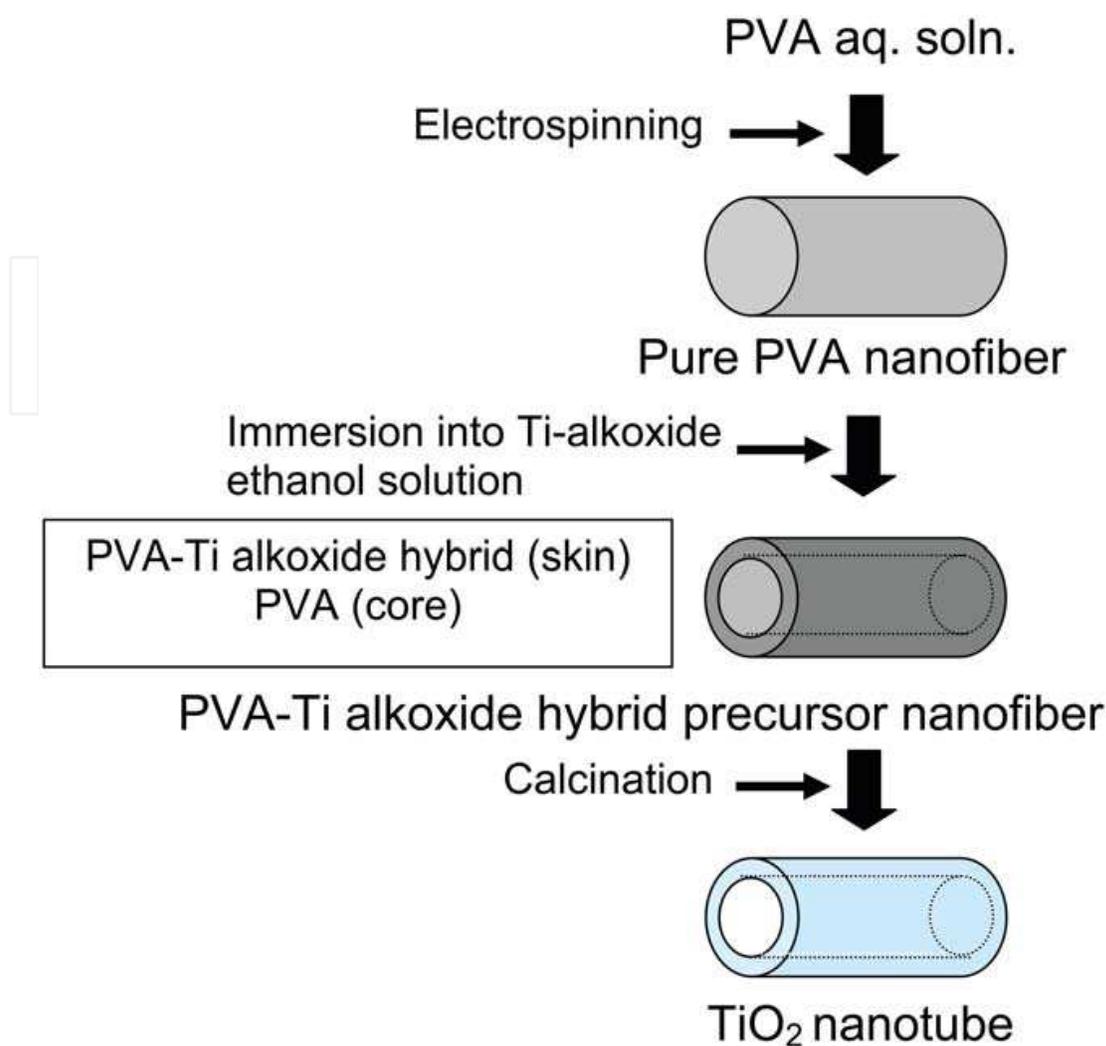


Fig. 8. Schematic illustration of the formation of TiO₂ nanotubes by our method.

Fig.10 shows the XRD curves of the residues (TiO₂) obtained by calcination of the precursor nanofibers at 400-800°C for 5 hours. Anatase type TiO₂ is mainly formed at 400-600°C, and the peak intensities increase with an increase in the calcination temperature. Rutile type (rutile-anatase mixed) TiO₂ is formed above 600°C.

Fig.11 shows the nitrogen adsorption isothermes (-196°C) of the TiO₂ nanotubes (calcination temperature range: 400-700°C). The adsorption amount of the TiO₂ nanotubes decreases with an increase of the calcination temperature. This is due to the sintering of TiO₂. The specific surface areas of the TiO₂ nanotubes were obtained from Fig.10 using the B.E.T. equation. The areas are 75.9 m²/g (calcination at 400°C), 38.8 m²/g (500°C), 17.4 m²/g (600°C) and 6.4 m²/g (700°C) (the area of pure PVA nanofiber was 3.8 m²/g). The average pore diameters of the TiO₂ nanotubes were 8.3 nm (400°C), 14.8 nm (500°C) and 21.4 nm (600°C). These pore sizes are not reflected the hollow size of the TiO₂ nanotubes, because the hollow size is several hundred nanometers. Therefore, it is likely that the TiO₂ nanotubes have mesopores on their nanotube wall (Fig.12). The mesopores would be through-holes formed by thermal decomposition of PVA in the precursor hybrid nanofibers. By the presence of the mesopores, the specific surface area of the TiO₂ nanotubes becomes larger, and the photocatalytic reaction using the TiO₂ nanotubes would occur effectively. The TiO₂ nanotube calcined at 700°C had a non-porous wall due to the sintering.

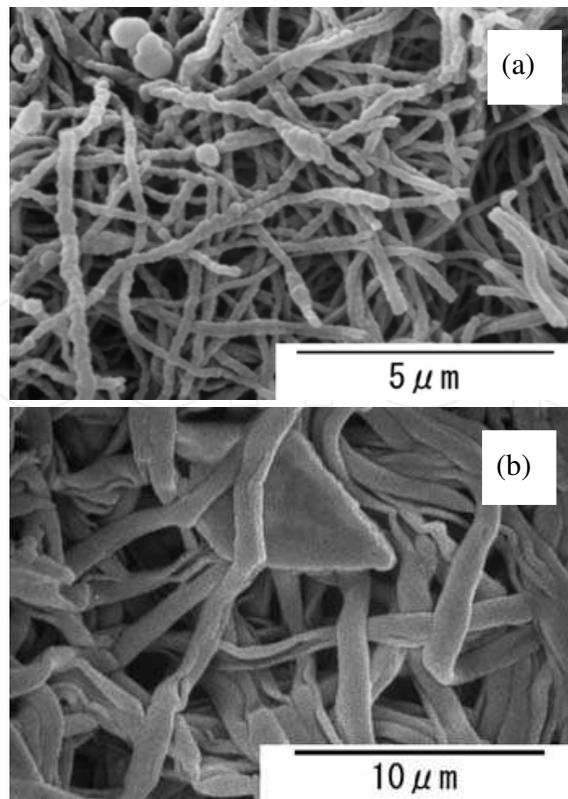


Fig. 9. SEM images of (a) non-hollow TiO₂ nanofibers and (b) wall-broken TiO₂ nanotubes.

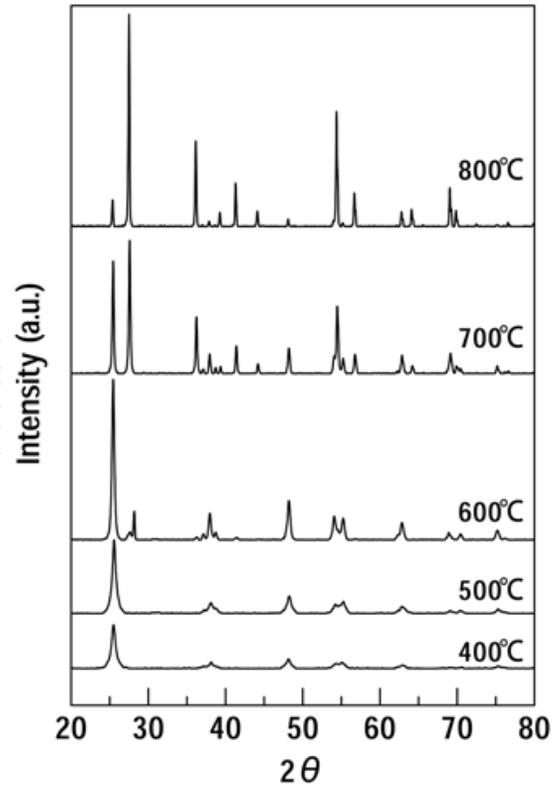


Fig. 10. XRD curves of TiO₂ nanotubes calcined at prescribed temperatures for 5 hours in air.

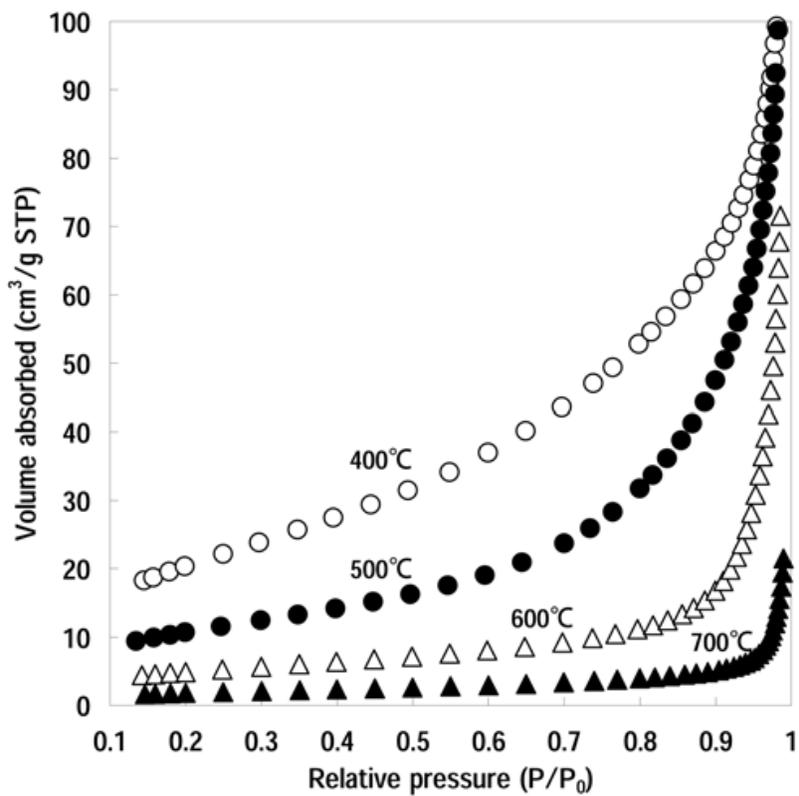


Fig. 11. Nitrogen adsorption isotherms (-196°C) of TiO₂ nanotubes calcined at prescribed temperatures for 5 hours in air.

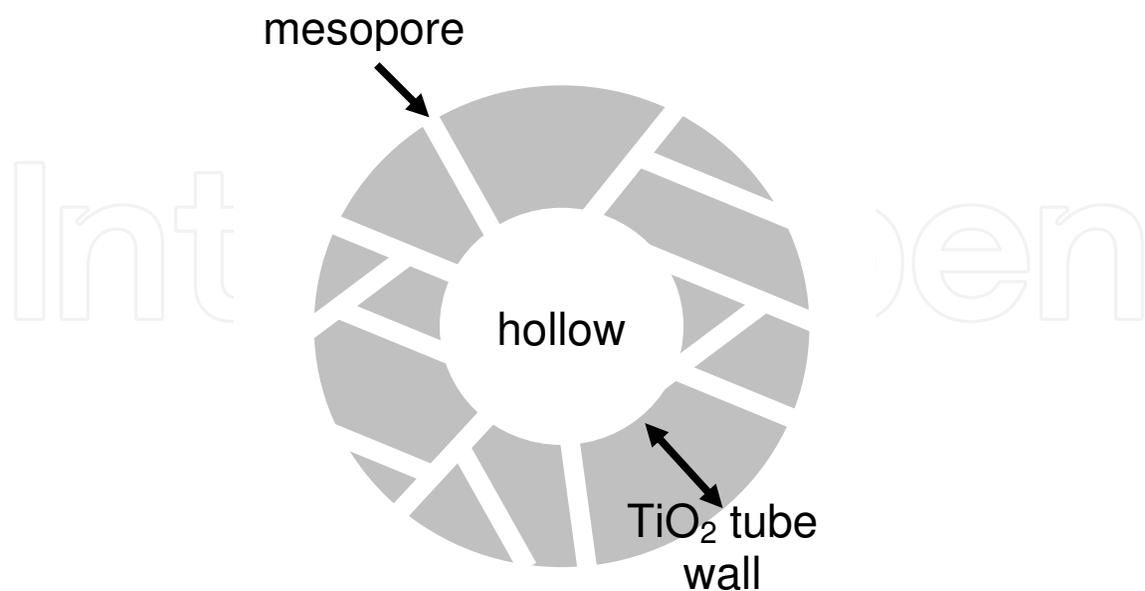


Fig. 12. Schematic illustration of TiO₂ nanotube obtained by our method (cross-section).

3.3 Photocatalysis of TiO₂ nanofibers and TiO₂ nanotubes

The photocatalysis of the TiO₂ nanofibers and nanotubes was investigated. Fig.13 (a) (b) show the relationship between the decomposition rate of methylene blue and the irradiation

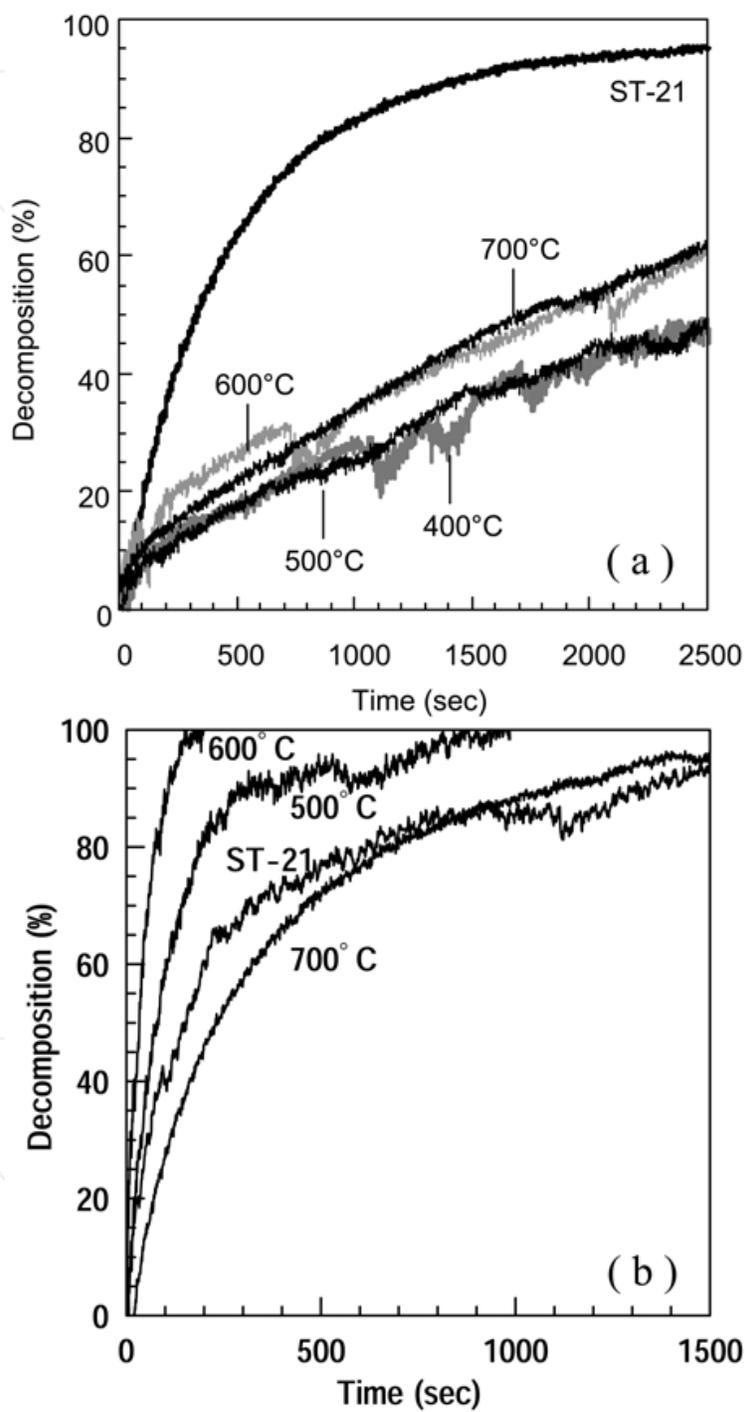


Fig. 13. Effect of irradiation time of white light upon the decomposition rate of methylene blue by the photocatalysis of TiO₂ nanofibers obtained by calcination of (a) PVA-TL and (b) PVA-TTIP hybrid nanofibers at prescribed temperatures.

time of white light for each TiO₂. Each figure includes the result of commercially available anatase type TiO₂ nanoparticles (ST-21, particle size: 20 nm, specific surface area: *ca.*50 m²/g, Ishihara Sangyo Kaisha, LTD. Japan).

In Fig.13 (a), the photocatalysis of the TiO₂ nanofibers calcined at 600°C and 700°C is higher than that of the TiO₂ nanofibers calcined at 400°C and 500°C, but the major differences between each TiO₂ nanofiber are not observed. The crystallinity of anatase-type TiO₂ increases with calcination temperature, though the specific surface area becomes lower. The photocatalysis of the TiO₂ nanofibers would be affected by both the crystallinity and pore characteristics of the TiO₂ nanofibers. The TiO₂ nanofibers have good photocatalysis, but the properties of these TiO₂ nanofibers are inferior to that of ST-21. The specific surface area of the TiO₂ nanofiber calcined at 400°C (56.4 m²/g) is higher than that of ST-21, but the ST-21 excels in photocatalysis. This would be due to the difference of the crystallinity of anatase.

In Fig.13 (b), the photocatalysis of the anatase type TiO₂ nanotubes (calcination at 500°C and 600°C) is clearly higher than that of ST-21, and the nanotube (calcination at 600°C) shows the highest photocatalysis in this experiment. Also, the photocatalysis of the rutile-anatase mixed TiO₂ nanotube (calcination at 700°C) is equivalent to that of ST-21. The specific surface area of ST-21 is higher than that of TiO₂ nanotubes, but the TiO₂ nanotubes excel in photocatalysis. At the present stage the reason why is uncertain, but the structure of the nanotube might contribute to the efficient photocatalysis of TiO₂. In other words, electron holes will be formed at the surface of the hollow when the TiO₂ nanotubes are irradiated. And the excited electron moves to inside the hollow because it might be the non-irradiation area (the electron density might be low). Thus the oxidation site and the reduction site would be separated, and the photocatalysis would proceed efficiently.

4. Conclusion

Titanium oxide (TiO₂) nanofibers were formed by calcination of poly(vinyl alcohol) (PVA)-Ti lactate hybrid precursor nanofibers in air. The fiber diameters of the PVA-Ti lactate hybrid nanofibers were 200-350 nm, and the fiber diameters of the TiO₂ nanofibers were 70-80% of those of the PVA-Ti lactate hybrid nanofibers. The specific surface area and average pore diameter of the TiO₂ nanofibers calcined at 500°C for 5 hours were 21.0 m²/g and 7.4 nm, respectively.

TiO₂ hollow-nanofibers (nanotubes) were formed by calcination of PVA-Ti alkoxide hybrid nanofibers. The outer and inner diameters of the TiO₂ nanotubes calcined at 500°C for 5 hours were *ca.*440 nm and *ca.*270 nm, respectively. The specific surface area of the nanotubes was 38.8 m²/g, and the existence of mesopores (average pore diameter, 14.8 nm) on the nanotube wall was indicated by the nitrogen adsorption isotherm (-196°C). The photocatalysis of the TiO₂ nanotubes was superior to that of commercially available anatase type TiO₂ nanoparticles.

The TiO₂ nanofibers and nanotubes have the advantage of being easily fixed on other materials, such as a refractory fabric, without a binder by the fiber length (TiO₂ powders such as ST-21 requires a binder in order to be fixed on other materials.). Fig.14 shows the SEM images TiO₂ nanofibers formed on carbon microfibers without a binder. These fibers are expected to be used as an air filter.

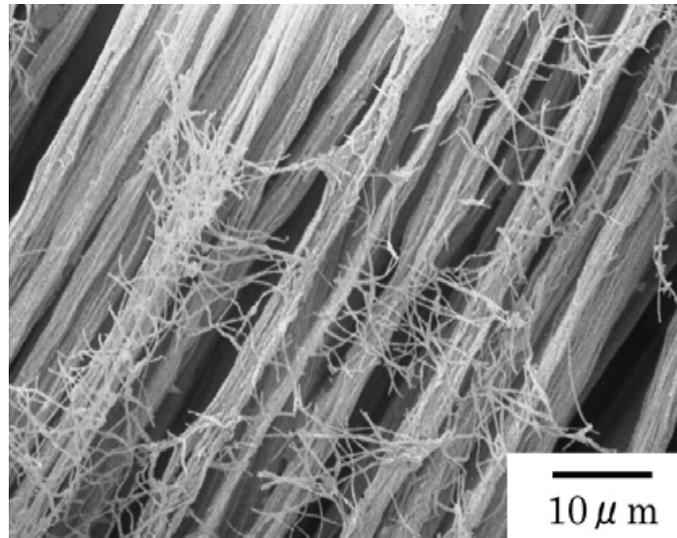


Fig. 14. SEM image of TiO₂ nanofibers formed on carbon microfibers (calcination temperature: 500°C).

5. Acknowledgements

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Nanofibers

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“There's Plenty of Room at the Bottom” this was the title of the lecture Prof. Richard Feynman delivered at California Institute of Technology on December 29, 1959 at the American Physical Society meeting. He considered the possibility to manipulate matter on an atomic scale. Indeed, the design and controllable synthesis of nanomaterials have attracted much attention because of their distinctive geometries and novel physical and chemical properties. For the last two decades nano-scaled materials in the form of nanofibers, nanoparticles, nanotubes, nanoclays, nanorods, nanodisks, nanoribbons, nanowiskers etc. have been investigated with increased interest due to their enormous advantages, such as large surface area and active surface sites. Among all nanostructures, nanofibers have attracted tremendous interest in nanotechnology and biomedical engineering owing to the ease of controllable production processes, low pore size and superior mechanical properties for a range of applications in diverse areas such as catalysis, sensors, medicine, pharmacy, drug delivery, tissue engineering, filtration, textile, adhesive, aerospace, capacitors, transistors, battery separators, energy storage, fuel cells, information technology, photonic structures and flat panel displays, just to mention a few. Nanofibers are continuous filaments of generally less than about 1000 nm diameters. Nanofibers of a variety of cellulose and non-cellulose based materials can be produced by a variety of techniques such as phase separation, self assembly, drawing, melt fibrillation, template synthesis, electro-spinning, and solution spinning. They reduce the handling problems mostly associated with the nanoparticles. Nanoparticles can agglomerate and form clusters, whereas nanofibers form a mesh that stays intact even after regeneration. The present book is a result of contributions of experts from international scientific community working in different areas and types of nanofibers. The book thoroughly covers latest topics on different varieties of nanofibers. It provides an up-to-date insightful coverage to the synthesis, characterization, functional properties and potential device applications of nanofibers in specialized areas. We hope that this book will prove to be timely and thought provoking and will serve as a valuable reference for researchers working in different areas of nanofibers. Special thanks goes to the authors for their valuable contributions.

How to reference

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