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Suttinart Noothongkaew, Ha Kyun Jung, Orathai Thumtan, Ki-Seok An

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Synthesis of free-standing anatase TiO₂ membrane by using two-step anodization

Suttinart Noothongkaew^{1*}, Ha Kyun Jung², Orathai Thumtan³,
and Ki-Seok An⁴

^{1,3}85 Sathonlamark Rd. Warin Chamrap, Department of Physics, Faculty of Science, Ubon Ratchathani University, 34190 Thailand

^{2,4}Korea Research Institute Chemical of Technology, Daejeon, 30-5343, South Korea

^asuttinart_n@yahoo.com

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Abstract. Free-standing titanium dioxide (TiO₂) membranes with highly ordered, vertically aligned nanotube arrays were fabricated by using two step anodization of pure titanium (Ti) foil in fluorine containing ethylene glycol. TiO₂ nanotubes were prepared under anodizing voltage of 50 V on Ti foil, followed by a thermal annealing at temperature of 200 °C for 10min, and 450 °C for 2h, respectively. Subsequently, anodization was carried out at high voltage of 70V which directly induced detachment of the nanotube membranes from Ti substrate. The film with high-quality surface and open-ended tubes could be obtained, which was easily peeled off and transferred onto other substrates. Free-standing TiO₂ membranes were characterized by field emission scanning electron microscopy (FE-SEM), X-ray diffraction (XRD), UV-vis spectroscopy, and X-ray photoelectron spectroscopy (XPS). As a result, we found that the diameter increased while tube length decreased with increasing concentration of DI water. Additionally, the thickness of film increased with increasing anodization time.

1. Introduction

Recently, TiO₂ nanotubes (NTs) are considered as prominent material due to their large surface area to volume ratio, long term stability, non-toxicity, low cost, and broad functionality. TiO₂ NT arrays have been extensively studied for their applications in sensors for hydrogen and oxygen gasses, humidity, solar cells, photocatalytic degradation, water photoelectrolysis, and so on [1-5]. However, a film of TiO₂ nanotube arrays on Ti foil substrate is opaque, thereby limiting the amount of light entering the surface of TiO₂. As a result, there is a decrease in photocatalytic activity. In addition, the presence of TiO₂ film on substrate becomes a barrier to the light response. In this paper, we report the synthesis of free-standing TiO₂ membranes that stand alone without the presence of a Ti substrate. The advantages of free-standing TiO₂ membranes are the decreasing of the barrier layer on the bottom of the tubes which enable electrons to transfer directly to the surface of TiO₂, and high stability to mechanical vibrations and easy to used or integrate with other substrates [6-20]. Thereby, several procedures have been proposed for the synthesis of free-standing TiO₂ membranes including ultrasonic splitting, chemically assisted delamination, solvent evaporation, and variation in applied voltages [10-20]. However, applications of free-standing TiO₂ in devices greatly depend on their crystallinity. In order to obtain crystallized TiO₂ arrays, the free-standing membranes will be annealed at high temperature. Normally, membranes curl and collapse into small pieces during the high temperature process. In this work, large scale, highly ordered nanotube array, non-curling, without any cracks or collapses of free-standing TiO₂ membranes were obtained by using two-step anodization. The free-standing TiO₂ nanotubes were considerably flexible which allowing for a variety of applications, and they could be attached onto foreign substrates.

2. Experimental details

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