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Electrophoretic Synthesis of Titanium Oxide Nanotubes

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P A P E R   I N F O

A B S T R A C T

In the current research project, sol-gel electrophoresis technique was utilized to grow titanium dioxide (TiO$_2$) nanotubes. A titanium sol was prepared using organometallic precursors of titanium to fill the template channels. The prepared sol was driven into nanopores of porous anodic aluminum oxide templates under the influence of a DC electric field to form nanotubes on the pore walls. Successful deposition on the template pore walls was achieved after broadening the pores through chemical etching using phosphoric acid. The simultaneous thermal analysis (STA) of the samples determined the onset temperature of the anatase TiO$_2$ crystallization. After calcination, the phase structure of the produced nanotubes was evaluated based on the X-ray diffraction (XRD) investigations. Scanning electron microscopy and energy dispersive X-ray (SEM and EDX) studies showed that TiO$_2$ nanotubes with uniform size and shape have been electrophoretically grown in the aluminum oxide template channels.

NOMENCLATURE

DC   direct current

JCPDS   Joint Committee on Powder Diffraction Standards

1. INTRODUCTION

Titanium dioxide or titania (TiO$_2$) is a well known metal oxide material which has been extensively used in pigment industry for several decades. This material is also known as an advanced material with various novel applications ranging from gas sensors to photoelectrochemical solar cells [1, 2]. It is, in fact, the most commercially used metal oxide semiconductor material because of its excellent mechanical and chemical stability and its rather low cost. This material has also significant advantages such as being chemically inert and resistant to chemical and sunlight corrosion. So, the TiO$_2$ devices can be used for long periods of time at ambient [3-5]. Besides, TiO$_2$ nanostructures are highly resistant to organic solvents, and TiO$_2$ has high electron-hole recombination time, compared to the other

metal oxide semiconductors, which are desirable in photocatalytic decomposition of organic materials. They also have the advantage of self-cleaning under ultraviolet radiation, strong oxidizing power of the generated holes and redox selectivity [6]. Therefore, nowadays, TiO$_2$ nanostructures are extensively used in solving environmental problems such as recycling water and sewage. TiO$_2$ has several well known crystalline polymorphs.

Among different TiO$_2$ polymorphs, the anatase TiO$_2$ phase shows more photocatalytic activity in ultraviolet light due to its enhanced electronic and optical properties. The nanostructured anatase-phase TiO$_2$ material has been investigated rigorously and found extensive applications in the last two decades [7-9].

Charge tranfer efficiency of a TiO$_2$ nanostructure depends on its geometry. Therefore, synthesis of one-dimensional nanostructures such as nanotubes and nanorods is usually considered because of their supreme capabilities in photoelectrochemical reactions due to the

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improved charge tranfer. In fact, different research teams have already shown that uniform one-dimensional TiO_2 nanostructures efficiently improve the energy conversion in the photocatalytic reactions. So, replacing the randomly oriented grains of the conventional TiO_2 meshes with well crystallographically oriented grains in a uniform array of either TiO_2 nanotubes or nanorods, which are grown out of the substrate, may improve the photocatalytic activity of the material.

The advantage of producing TiO_2 nanotubes is their high surface area. Higher surface area can guarantee higher catalytic activity. Therefore, TiO_2 nanotubes are suitable one-dimensional materials for nanoscale catalysis. This is why many attempts have recently been carried out and reported in the literature to produce TiO_2 nanotubes and offer new applications for the produced nanotubes [10-20]. Various methods have been utilized to produce TiO_2 nanotubes including anodic oxidation of titanium foils [11-13], photoelectrochemical etching [16-17], sol-gel processes [18-19], and hydrothermal synthesis [20].

Fabrication of TiO_2 nanotube arrays by direct anodization of titanium foils was reported for the first time in 2001 by Grimes and co-workers, as reported in Gong et al., [12]. Since then, this method has been extensively studied and employed in producing TiO_2 nanotubes because of its simplicity [21]. However, technological use of these anodized nanotubes is limited due to the wide band gap of pure titania (i.e., 3.2 eV) which can efficiently absorb the UV section of the radiated solar energy via the electron-hole pair generation mechanism. But, UV light is just about 8% of the sun's energy compared to visible light (45%). Therefore, shifting the optical response of TiO_2 nanotubes from the UV light towards full solar spectrum is highly desirable for improving their photocalytic activity.

Many attempts have already been reported in the literature to shift the optical response of anodized TiO_2 nanotubes toward the visible light by doping these nanotubes with transition metal cations. But, this approach often decreases electron-hole recombination time, which affects the photocatalytic efficiency of TiO_2 material, and deteriorates the chemical stability of the nanotubes [16].

As a suitable alternative, template-based methods have been the focous of attention for producing TiO_2 nanotubes. These methods, in principle, consist of three procedures: Fabricating a nanoporous template such as a nanoporous aluminum oxide template, pore filling with the precursor solution and selective etching of the template. Nanoporous aluminum oxide templates are fabricated via anodic oxidation of aluminum in an electrochemical bath. This technique forms a porous oxide layer over the aluminum substrate. Aluminum oxide templates have several advantages which make

them a basis for nano-patterning in nanotechnology. Porous aluminum oxide templates can be fabricated from either bulk or thin aluminum substrates such as deposited aluminium layers on the desired substrates depending on the application. The template pores in these materials are in principle in nanometre scale. The template pores can have high aspect ratio and highly regular nature. The geometric coefficients of the pores such as pore size and aspect ratio can be tuned over large areas by varying the anodic oxidation parameters. Therefore, it is possible to produce uniform TiO_2 nanotubes with controlled size and aspect ratio using these templates. Moreover, anodic aluminum oxide membranes offer the advantage of possible in-situ annealing of the deposited oxide materials within the template pores because of the refractory properties of aluminum oxide.

Other useful properties of aluminum oxide templates include optical transparency over a wide spectral range, chemical stability and low cost. Besides the mentioned advantages, they are potential support mateials for TiO_2 catalysts because it is already reported that coating TiO_2 photocatalyst films on Al_2O_3 improves the photocatalytic activity of the grown films [22].

To produce titanium oxide nanotubes in nanoporous aluminum oxide templates, different techniques are already used for the pore filling stage such as vacuum assisted sol-gel and template wetting by the precursor sol [23-24].

In this study, we employ sol-gel electrophoretic deposition technique to fill the aluminum oxide template pores. It is well known that a colloidal sol contains surface charged nanoclusters, with the desired stoichiometry, which are generated by the hydrolysis and condensation reactions during sol preparation.

This surface charge interacts with other charged species in the sol (i.e., ions) to produce a double-layer charged structure around the suspended particle. These layers are generally called the diffusion (Gouy) and Stern layers. Gouy layer is composed of ions which are tightly bonded to the surface of the cluster, but in the Stern layer, ions are not firmly bonded to the surface and can be replaced by similarly charged ones. Electrostatic stabilization of the sol is based on this double-layer charged structure which prevents particles from approaching each other. In electrophoretic deposition technique, an external electric field is applied to such an electrostatically-stabilized solution.

The externally applied electric field sets these ion surronded charged particles in motion toward the conductive electrodes which are inserted in the sol and, therefore, could be used to deposit the suspended particles on a substrate. This technique is schematically illustrated in Figure 1.

Sol-gel electrophoretic deposition has already been used to produce thin or thick films of different

compositions and has shown to yield layers with greater thickness, density and quality than the traditional sol-gel method alone. Besides, this technique has been utilized to produce one-dimensional nanostructures of complex oxides [25]. But, in the present work, it is utilized to produce TiO_2 nanotube arrays by a template-based method using nanoporous anodic aluminum oxide templates.
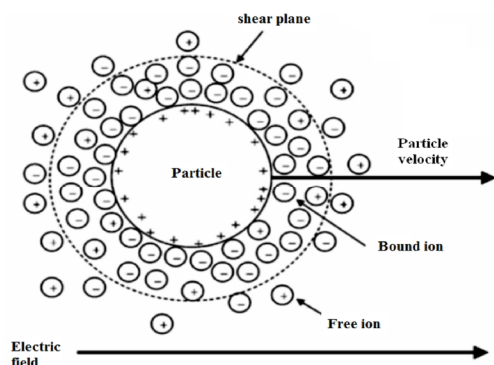

Figure 1. Schematic illustration of sol electrophoretic deposition technique: simultaneous migration of a suspended particle and its double-layer structure in the solution due to an applied electric field.

2. EXPERIMENTAL

2. 1. Materials　　The following materials were used in this project to prepare porous aluminum oxide templates and TiO_2 nanotubes: Al foils, phosphoric acid, chromic acid, titanium (IV) n-butoxide, glacial acetic acid, acetyl acetone, ethanol, ethylene glycol. All of the mentioned materials were in analytical grade and purchased from Merck Inc. Double distilled water, comercially available pure aceton, sulfuric, hydrochloric and nitric acid were also used.

2. 2. Porous Aluminum Oxide Templates　　The first part of this research deals with the production of porous aluminum oxide templates for the growth of nanotubes through two-step anodizing of pure aluminum foils. The Al foils were first cleaned in methanol and acetone, respectively, under ultrasonic vibration, and their surfaces were chemically cleaned in a molar mixture of 10:20:70 of HCl, HNO_3 and H_2O. They were then annealed in nitrogen ambient at 500°C for 5 hours to increase the grain size and provide better homogeneity for uniform development of the pores. The foil surfaces, after heat treatment, were electrochemically polished using a mixture of water solutions of H_3PO_4 and H_2SO_4 to decrease their surface roughness caused by rolling down to the submicron

scale. Electropolishing improved the surface roughness from about one micrometer down to about 30 nanometers in an area of $5 \times 5 \mu m^2$, as determined by atomic force microscopy. The polished aluminum sheets were placed on a stainless steel plate to be exposed to a 10 wt. % aqueous solution of phosphoric acid and anodized in a cooled electrochemical cell, held at a constant temperature of 1°C. A DC electric field was applied for anodizing. The first anodized layer was subsequently removed in a mixture of phosphoric acid (6 wt. %) and chromic acid (1.8 wt. %) solutions. So, a patterned aluminum surface was prepared for uniform growth of template pores in the second anodizing step [24]. A constant DC electric potential of 100V was applied to the anodizing cell in both of the anodizing steps. After second anodizing, the unanodized aluminum substrate was removed in a saturated solution of $HgCl_2$ to achieve a transparent anodic oxide membrane.

2. 3. Sol Preparation and Deposition　　A stable sol was prepared by hydrolysis of titanium (IV) n-butoxide as the titanium precursor. In order to control the kinetics of the hydrolysis reaction, titanium n-butoxide (19 ml) was added to glacial acetic acid (22.9 ml) first to chemically modify the hydrolysis-condensation reactions. Double distilled water (9 ml), acetyl acetone (5.2 ml), pure ethanol (51.2 ml) and ethylene glycol (3.1 ml) were also added to the prepared solution after that procedure. Water was added to hydrolyze the alkoxide material. It was added dropwise, under mechanical stirring, to avoid rapid formation of the gel. The final solution was magnetically stirred for 24 hours and stored in sealed containers filled with argon gas. The resulting sol was transparent and stable for several months.

To have a deposition template, the nonconductive barrier layer of the prepared templates was chemically opened and the pores were simultaneously broadened after etching the templates with a 5 wt.% phosphoric acid solution. The chemical etching of the nonconductive barrier layer was a longtime process because the layer is composed of a dense and pure Al_2O_3 material. This procedure took more than two hours at room temperature.

DC electrophoresis technique was employed to fill the template channels with the prepared titanium sol. To prepare a deposition electrode for DC electrophoresis, the bottom of the templates was first coated by sputtering a 100-nm thick metal film using either gold, silver, aluminum or copper. The metalized template was fixed in a specifically designed stainless steel holder to be connected to the negative pole of a DC source-meter instrument during the deposition process. Thus, a so-called working electrode was produced. Another

stainless steel plate was also used as the counter electrode. The deposition process was carried out at room temperature, and any change in the electrophoretic current was recorded by the digital source-meter.

After the deposition was complete, the filled template was removed from the working electrode, and the excess sol on its surface was wiped away mechanically. The filled templates were subsequently dried at 110°C for 12 hours to evaporate unbounded water and solvent. During this step, the deposited nanotubes form gelled layers on the pore walls. Then, the template containing the deposited nanotubes was heat treated. The heat treatment step was carefully monitored to crystallize only anatase TiO_2 phase from the amorphous nanotubes.

2. 4. Characterization   Thermal behavior of the prepared titanium gel was investigated by a Pyris Diamond (SII) simultaneous thermal analysis (STA) system. The heat treatment process were evaluated by a Philips PW 3040 X-ray diffraction (XRD) system using CuKα radiation to examine formation of the desired anatase phase. The mean crystallite size, for each heat treatment condition, was calculated from the XRD data based on the Scherer equation. Formation of nanotubes as well as size and morphology of the grown nanotubes were observed on a Philips XL 30 scanning electron microscope (SEM) system. An energy dispersive X-ray (EDX) spectrometer was also used to analyze composition of the grown nanotubes. A fixed electrophoretic voltage was applied and current intensities were simultaneously measured using a Keithley 2400 LV digital source-meter.

3. RESULTS AND DISCUSSION

3. 1. Titanium Oxide Nanotubes Figure 2 shows the SEM planar view of an array of TiO_2 nanotubes. The observed nanotubes have been deposited in the channels of a nanoporous anodic aluminum oxide template using DC electrophoretic technique. To see this array, the filled template, after heat-treatment, has been mounted on a glass sheet to be polished by nano-alumina powder and subsequently removed by chemical etching. Template etching has been done in a 5 wt.% caustic soda solution at room temperature for about 15 minutes. The etched template was subsequently rinsed with deionized water and dried.

It is clearly evident in Figure 2 that DC electrophoresis of the prepared sol has resulted in a hollow tubular structure. The nanotubes have stuck out of the template after etching. The tube walls are observed in this figure without any hindrance. Although the template shown in Figure 2 has been heavily etched, the separating walls between the tubes, in some areas,

can be distinguished. Figure 2 also shows that nearly all of the channels are filled with an array of TiO_2 nanotubes. The micrograph shows the nanotubes deposited at a voltage of 3V for 10 minutes. The average outer diameter of the tubes is about 180 nm which is consistent with the internal diameter of the used template pores. The average thickness of the tubes was also measured using the scale bars on the SEM images to be about 45 nm.

To evaluate the suitability of DC electrophoretic deposition technique in producing tubular TiO_2 arrays, larger areas of the filled template were also investigated. Figure 3 shows a larger area of the same sample as in Figure 2 at a lower magnification. It is observed that nearly all of the channels are filled, and the grown TiO_2 array has covered the whole visible area of the template which shows good filling efficiency of the porous aluminum oxide template with TiO_2 nanotubes. The hollow nature of the array indicates that a tubular rather a rod or wire-like structure is formed throughout. This reveals the suitability of the DC electrophoresis as an effective method for producing close-packed arrays of TiO_2 nanotubes through a template-based process.
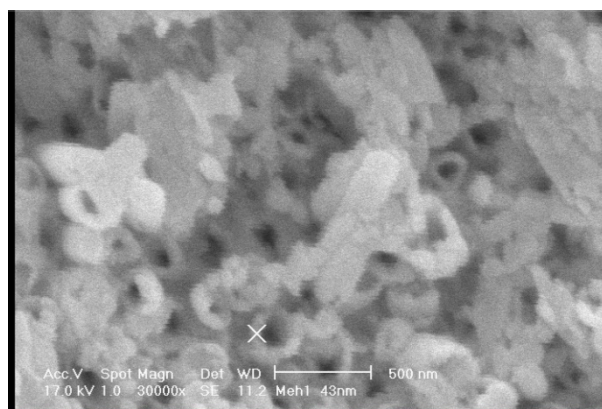

Figure 2. SEM micrograph of an array of TiO_2 nanotubes grown by DC electrophoretic technique.
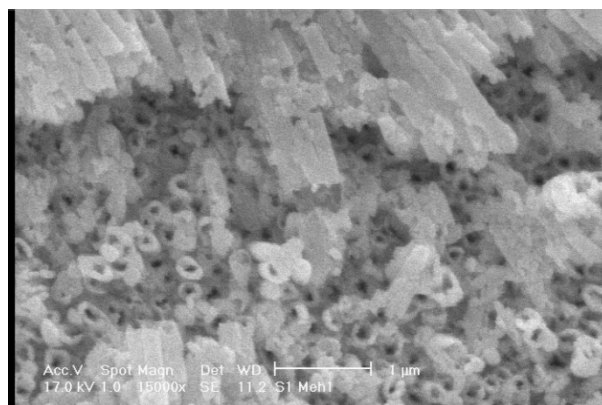

Figure 3. SEM micrograph of the array of TiO_2 nanotubes of Figure 2 at a lower magnification.

The nonconductive barrier layer of the prepared templates were chemically opened with a 5 wt.% phosphoric acid solution. Therefore, the possibility of tubular growth of one-dimensional TiO_2 nanostructures within the template channels reveals the conductive behavior of the remained anodic oxide layer. It has already been reported that this barrier layer consists of an n-type semiconductor aluminum oxide layer at the electrolyte interface and a dielectric passive layer at the Al electrode.

3. 2. Energy Dispersive X-Ray (EDX) Analysis
EDX analyses were carried out to evaluate the composition of the produced nanotubes qualitatively. Figure 4 shows the EDX spectrum of the marked nanotube of Figure 2. Since the filled template was thin due to mechanical polishing and chemical etching, the spectrum represents the X-ray lines from the nanotubes as well as some lines originating from the bottom surface of the template. The Cu lines are originating from the coated surface of the template which was metalized after barrier thinning. The aluminum peaks are from the remained porous template. Moreover, the top surface of the sample has been coated by a thin gold layer to avoid electric charging during microscopic investigations. Therefore, Au lines were expected in the EDX spectrum. Taking these considerations into account as well as heat treatment in an oxygen-rich atmosphere, we can conclude that the analyzed nanotubes, grown in an aluminum oxide template, are composed of titanium oxide.

3. 3. Simultaneous Thermal Analysis   Figure 5 shows the result of the simultaneous thermal analysis (STA) of the dried titanium gel. In the differential thermal analysis (DTA) graph, an exothermic region is seen around the 294ºC peak which is also close to the 302ºC peak in the DTG graph. Since the later peak occurs after a steep decrease of the weight in the TG plot, both of 294ºC and 302ºC peaks are due to the exothermic decomposition of organic matter contained in the gel [26]. Later, another peak is in the DTG graph in Figure 5 at about 400ºC which follows with an exothermic peak in the DTA plot at 400ºC. This peak also follows with a steep decrease of the weight in the TG plot of Figure 5. Therefore, the discussed peaks are probably due to exothermic oxidation of the residual carbon species and formation of the amorphous titanium dioxide powder. However, the exothermic region which is seen around the 440ºC peak in the DTA plot is not close to any peak in the DTG graph. Moreover, this peak occurs in the region in which only small decrease in the weight is observed in the TG plot. So, one can conclude the discussed peak is the temperature that crystallization of the anatase titanium dioxide phase starts [26]. Later, due to evaporation of chemisorbed

water, a slow-rate decrease is observed in the TG graph after about 850ºC which is accompanied with a peak in the DTG graph at about 950ºC.

3. 4. X-Ray Diffraction Analysis   X-ray diffraction (XRD) analyses were performed to evaluate both the composition and phase structure of the titanium gel after firing. Because of presence of carbon-rich organometallic precursors, a special firing schedule was used in the sintering step. The samples were dried at about 110°C for 10 hours to ensure a complete evaporation of the organic solvent. They were subsequently annealed at 450°C for 2 hours to ensure complete burnout of the organic species and formation of the desired anatase phase. The samples were then furnace cooled down to the room temperature in air ambient.
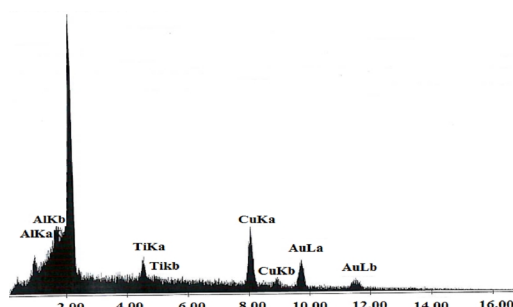

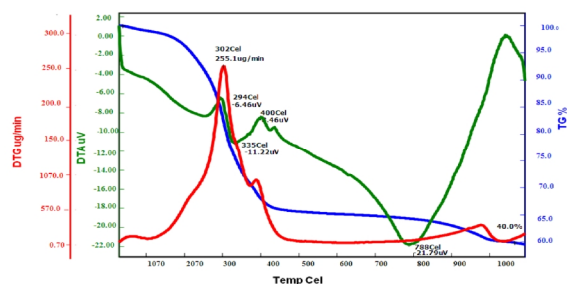**Figure 4.** The EDX spectrum of the marked nanotube in Figure 2.

Figure 5. Simultaneous thermal analysis (STA) of the dried titanium gel.
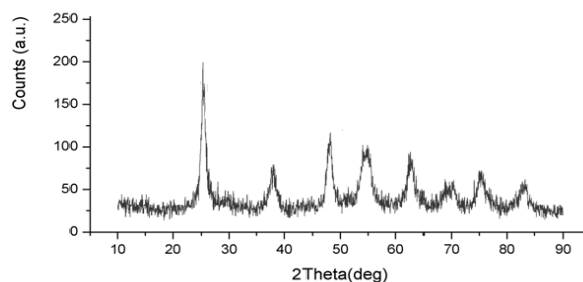

Figure 6. XRD pattern of the heat-treated TiO_2 gel at 450°C for two hours. All of the lines match the anatase phase.

Figure 6 shows the XRD pattern of the heat-treated TiO_2 gel produced as discussed. All peaks were identified according to their JCPDS standard cards. Since the gel was heat treated at 450°C, it is possible for both brookite and anatase phases to be present simultaneously. Since one-dimensional anatase nanostructures are very useful in photocatalytic applications, the heat treatment conditions were carefully tuned to avoid formation of undesirable brookite phase in the deposited nanotubes during firing. As can be seen in Figure 5, all the reflections belong to the anatase TiO_2 phase. Therefore, single-phase anatase TiO_2 nanotubes are achieved after the mentioned heat treatment condition.

The mean size of the synthesized anatase TiO_2 crystallites was calculated based on the Scherer equation. The calculation indicated that the heat treatment temperature (i.e., 450°C) has caused the mean crystallite size to increase to 16 nm.

5. CONCLUSION

- ❖ In this research, for the first time, TiO_2 nanotubes were successfully grown within the channels of porous anodic aluminum oxide templates by sol-gel electrophoretic technique. A DC electric field was applied to fill the template pores with the titanium sols.
- ❖ Highly stabilized titanium sols were prepared through chemical modification of titanium tetra-butoxide with glacial acetic acid.
- ❖ SEM micrographs indicated the suitability of the DC electrophoresis as an effective method to fill the aluminum oxide templates with TiO_2 precursor material.
- ❖ Close-packed arrays of TiO_2 nanotubes were produced after heat treatment of the filled templates.
- ❖ The possibility of tubular growth of one-dimensional TiO_2 nanostructures within the template channels confirmed the semiconducting behavior of porous anodic aluminum oxide layers formed on pure aluminum substrates. So, taking their chemical stability into consideration, these layers could be promising oxide semiconductors for future electronic applications.

6. ACKNOWLEDGMENT

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چکیده

در این پروژه، از تکنیک الکتروفورزیس (برق رانی) سل ژل برای رشد نانولوله های اکسید تیتانیم (TiO₂) استفاده گردید. سل حاوی پیش مواد ارگانومتالیک تیتانیم تهیه گردید تا به کمک آن انباشت نانولوله ها درون حفرات قالب گیرد. انباشت سل به درون نانوحفرات قالب با اعمال میدان الکتریکی مستقیم انجام گردید تا نانولوله ها بر روی دیواره حفرات تشکیل شوند. رشد موفقیت آمیز بر روی دیواره حفرات قالبها پس از فراخ کردن حفرات به کمک سونش شیمیایی با اسید فسفریک امکان پذیر گردید. با انجام آنالیز حرارتی همزمان (STA) دمای تبلور فاز مطلوب اکسید تیتانیم آناتاز در نانولوله های رشد داده تعیین و پس از انجام عملیات حرارتی، ساختار فازی نانو لوله‌های تولید شده به کمک بررسیهای پـراش اشـعه ایکس (XRD) ارزیابی گردید. بررسیهای میکروسکوپ الکترونی روبشی (SEM) و آنـالیز انـرژی پاشـیده اشـعه ایکس (EDX) نشان دادند که به این طریق آرایه‌هایی از نانولوله‌های TiO₂ با ابعاد و ساختار نسبتاً یکنواخت را می‌توان به روش برق رانی درون قالبهای اکسید آلومینیوم رشد داد

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