

Synthesis and characterization of TiO₂ nanotubes from electrochemical oxidation of titanium substrate

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Abstract – In the present work, an anodization synthesis of TiO₂ nanotubes using NaF aqueous solution is described. The anodized TiO₂ film samples on titanium foils were characterized by scanning electron microscopy and Raman spectroscopy. The morphological differences of the samples obtained were observed as a function of the experimental conditions, such as concentration and voltage during 4 h. The Raman spectroscopy studies indicated presence of nanostructures characteristic of the anatase phase of TiO₂ nanotubes.

Anodized titanium-oxide containing highly ordered, vertically oriented TiO₂ nanotube arrays is a nanomaterial architecture which can be applied in hydrogen generation, nanoelectronics and other applications. The anodic oxidation or oxidation by micro-arc (OMA) is a method for the formation of an oxide film on the surface of the anode. The phenomenon of lightning discharges associated with the process of electrolysis was discovered in the nineteenth century and was studied in detail in 1930 by Sluginov and by Gunterschultze and Betz. However, the benefits of this technology were explored only in the 1960's, when McNiell and Gruss used electrical discharges on cadmium niobate to deposit an electrolyte containing the ion niobium on a cadmium anode. Markov and co-workers performed the deposition of aluminum oxide anode under electric discharges conditions in the 1970's [1].

In the present work, samples of titanium plates of 5 cm x 3 cm were used and prepared by polishing both sides with SiC sandpapers (from 220 to 1200 mesh) and cleaned during 30 minutes under ultrasound (10 N HCl) and another 30 minutes under ultrasound (in acetone PA). A DC electrical source was used to generate the electrical potential required for TiO₂ growth nanotubes. A one compartment Teflon cell was used with a platinum counter electrode and the titanium sample as the working electrode.

Table 1 lists the experimental conditions used to synthesize the TiO₂ nanotubes.

Table 1. Synthesis parameters of the TiO₂ nanotubes

Sample	Electrolyte	Concentration	Voltage	Time
A	NaF	0,18M	25V DC	4h
B	NaF	0,18M	30V DC	4h
C	NaF	0,09M	25V DC	4h

Figures 1 (a), (b) and (c) show the surface morphology of samples A1, A2 and A3, in which it is possible to observe morphological differences due to the syntheses conditions employed for each sample. Figure 1(c) shows greatest average inner diameter and a more regular nanotube distribution than (a) or (b), indicating that a higher voltage is necessary and a low concentration of NaF favors a higher inner diameter.

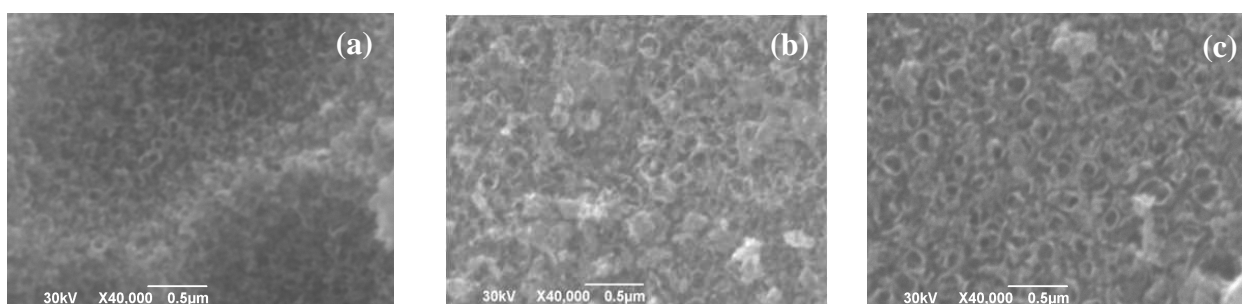


Figure 1: SEM topography of TiO₂ nanotube arrays obtained by anodization.

TiO₂ nanotube arrays obtained directly by anodization (without annealing) were analyzed by Raman spectroscopy and these studies showed the presence of TiO₂ nanostructures. The peaks at 400, 520 and 630 cm⁻¹ were associated to anatase phase of TiO₂ nanotubes [2].

References

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