Study of anodizing mode effects on synthesis of self-ordering TiO₂ nanotubes

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TiO₂ nanotubes were obtained by Ti anodizing in ethylene glycol solution. TiO₂ foil of 99.5% purity, 250 μ m thickness, purchased from AlfaAesar was used in the experiments. The electrochemical set-up consisted of an electrolysis power supply consort EV 243 and a classical two electrode cell. A sample was positioned vertically between two stainless steel plates as cathodes at a distance of ~ 13 mm from each other. A one-compartment thermostat bath was applied for Ti anodizing. The anodizing solution was stirred continuously using a magnetic stirrer and its temperature was fixed between 5 and 40 °C by thermostat. The anodizing electrolyte consisted of 0.1, 0.3 and 0.5 wt.% ammonium fluoride (NH₄F) in ethylene glycol + 2.0 vol. % H₂O. The current-time curves for the anodizing were recorded by means of a computer. The morphology, diameter and length of TiO₂ nanotubes were determined by scanning electron microscopy (SEM). The cross-section images were taken from cracked layers after bending of the samples. The outer diameter and length of the nanotubes reported were the average over at least five locations.

The effect of parameters such as applied voltages, anodizing time, electrolyte temperature and pH of electrolyte on the outer diameter of nanotubes and length of TiO_2 nanotubes were investigated. In the experiments the following parameters were used: applied voltage from 30 to 60; electrolyte temperature from 5 to 40 °C; 6, 7 and 8 for pH of electrolyte and 3, 6 and 10 hours for anodizing time. SEM study showed that length and diameter of the synthesized nanotubes were in the range of approximately 9–70 μ m and 80–170 nm respectively.

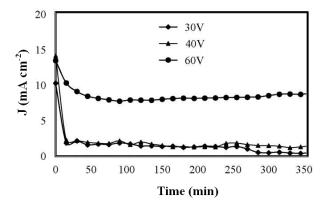


Fig. The current–time curves for Ti anodizing in ethylene glycol solution +NH₄F +2 vol.% water at constant voltage of 30, 40 and 60 V

Based on our results, nanotube length increased with increasing voltage, temperature and anodizing time. The outer diameter of nanotube increased also with increasing voltage and temperature, whereas outer diameter of nanotubes decreased significantly with increasing pH and no considerable change was observed with increasing anodizing time.

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