

ANODISED TiO₂ NANO-TUBES: VOLTAGE RAMP INFLUENCE ON THE OXIDE MORPHOLOGY AND INVESTIGATION OF PHASE CHANGES IN THE CRYSTAL STRUCTURE PROMOTED BY THERMAL TREATMENTS

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Abstract

The generation of titanium dioxide nano-tubes (Fig.1 and 2) by anodising commercially pure (99.6%) titanium in a fluorine-based electrolyte has been studied. Experiments have been performed using an electrolyte consisting of a 1M solution of sodium sulphate, with a small amount of sodium fluoride (0.1-1%wt) added. The electrolyte pH was controlled by addition of sulphuric acid, helping reduce unwanted precipitates that partially covered the nano-layer. The use of an initial voltage ramp prior to applying a constant voltage to the cell, caused a different morphology to develop in the anodised oxide nano-structure. Changes in the thickness of the oxide layer were also promoted by use of a controlled voltage ramp. The thermal stability of the nano-tubes layer has been investigated by annealing at different temperatures, from 200 to 600°C, for 3h. Scanning Electron Microscopy (SEM) indicates the nano-tube morphology is stable up to 500°C. X-Ray Diffraction (XRD) has been performed to monitor phase changes in the crystal structure developed by the thermal treatments. The as-prepared nano-tubes are amorphous, the anatase phase start to form at 300°C whilst the rutile phase is found above 500°C. Initial results obtained by characterising the as-prepared nano-tubes with Transmission Electron Microscopy (TEM) confirm the results and clearly show the three dimensional network which forms the structure. Electron diffraction at 200kV confirms the amorphous nature of the as-formed TiO₂ film.

Figures:

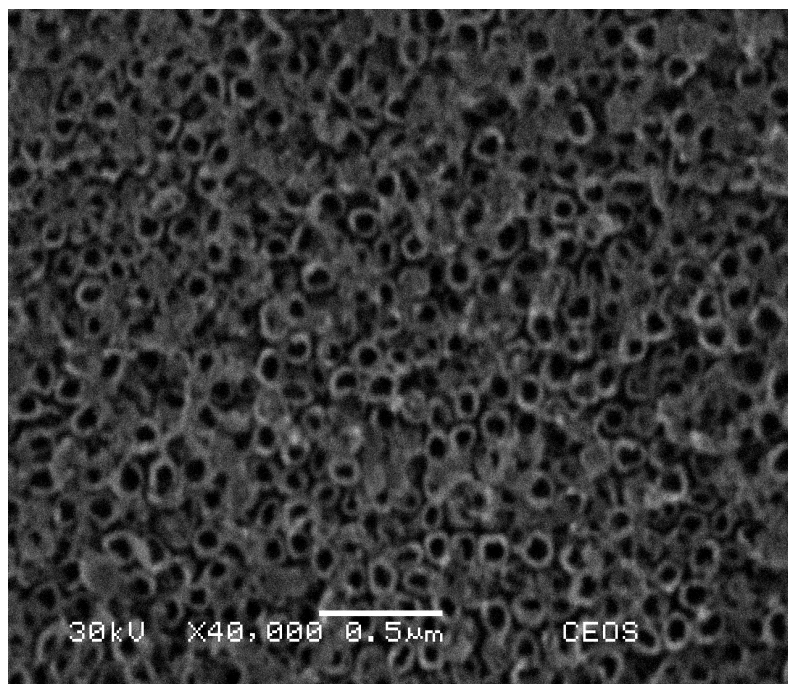


Figure 1. Top-view of a titanium dioxide nano-tubes array grown by anodisation in a fluorine-based electrolyte.

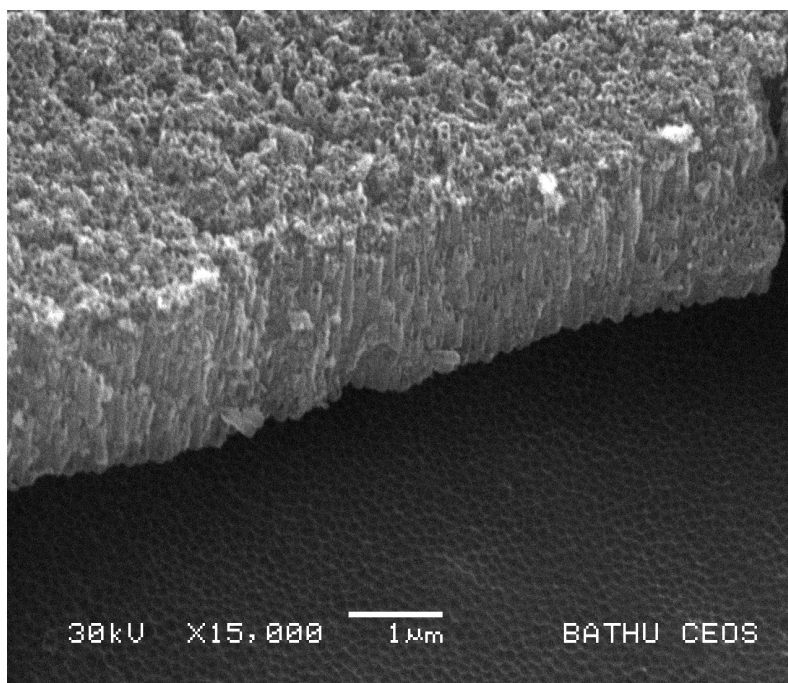


Figure 2. Cross sectional view of the nano-tubes layer. The concave-shaped layer of titanium underneath the nano-tubes is also shown.