



## Self-organized porous TiO<sub>2</sub> and ZrO<sub>2</sub> produced by anodization

Hiroaki Tsuchiya, Jan M. Macak, Andrei Ghicov,  
Luciano Taveira, Patrik Schmuki \*

*Department of Materials Science, Institute for Surface Science and Corrosion,  
University of Erlangen-Nuremberg, Martensstrasse 7, D-91058 Erlangen, Germany*

Available online 1 September 2005

---

### Abstract

The present work investigates the electrochemical formation of self-organized high aspect ratio TiO<sub>2</sub> and ZrO<sub>2</sub> nanotube layers. The formation and growth of a self-organized porous layer can be achieved directly by anodization without any templates in fluoride containing electrolytes. The morphology of the porous layers is affected by the electrochemical conditions such as the electrolyte composition, the pH and the exact polarization treatment (such as the potential sweep rate from the open-circuit potential to the anodizing potential). For Ti, nanotube layers are formed with diameters varying from approx. 20 nm to 100 nm and lengths from approx. 0.25 μm to 2.5 μm depending on the electrolytes and pH. On the other hand, for Zr, tubes of 50 nm in diameter and up to approx. 17 μm in length can be grown—a key parameter in this case is the potential sweep rate. The large difference between Ti and Zr in the achievable thickness of nanotube layers indicates a difference in the growth mechanism which may be based on the different chemical dissolution rates of electrochemically formed oxides.

© 2005 Elsevier Ltd. All rights reserved.

*Keywords:* Self-organization; Nanotube; Titanium oxide; Zirconium oxide; Anodization

---

---

\* Corresponding author. Tel.: +49 9131 852 7575; fax: +49 9131 852 7582.  
E-mail address: [schmuki@ww.uni-erlangen.de](mailto:schmuki@ww.uni-erlangen.de) (P. Schmuki).

## 1. Introduction

Nanoscale structuring of materials by exploiting localized dissolution (controlled localized corrosion) reactions has attracted a lot of interest in the recent years. Under optimized electrochemical conditions (applied potential, electrolyte composition, treatment, etc.) self-assembly or self-organization phenomena on the nanoscale can be initiated and exploited. In particular, porous self-organized  $\text{Al}_2\text{O}_3$  with a high aspect ratio pore geometry has been intensively investigated [1–3]. While considerable scientific thrust is directed towards the elucidation of the principles of the self-organized phenomena, technological efforts target applications, for example, for sensing [4] or for photonic crystals [5,6]. In addition to direct applications, the nanostructures have been also used as a template for the deposition and fabrication of secondary nanostructures such as metals [7], semiconductors [8] or polymers [9]. For other metals than Al, such as Ti [10,11], Zn [12] and Sn [13], only a little information was available on the formation of porous structures until a short time ago. Recently, we have reported the formation of self-organized porous structures on a series of valve metals such as Ti [14–18], Zr [19–21], Hf [22], Nb [23], Ta [24] and W [25] in fluoride-ion containing electrolytes while only a compact oxide is formed in fluoride-free electrolytes. In order to achieve pore formation on such valve metals the dissolution of electrochemically formed oxides as soluble complexes is required. Fluoride ions are expected to work as dissolution agents—to dissolve the formed anodic oxide by forming soluble valve metal fluoro-complexes.

In the present work we report the formation of high aspect ratio self-organized  $\text{TiO}_2$  and  $\text{ZrO}_2$  nanotube layers and show how electrochemical conditions during anodization can affect the morphology and structure of the layers.

## 2. Experimental

### 2.1. Materials

Ti (99.6% purity, Advent Research Materials, England) and Zr (99.8%, Goodfellow, England) samples with 0.1 mm thickness were degreased by sonicating in acetone, isopropanol and methanol, followed by rinsing with deionized water and drying in a nitrogen stream.

### 2.2. Electrochemical treatment

Samples were contacted and then pressed against an O-ring in an electrochemical cell, leaving  $1\text{ cm}^2$  exposed to an electrolyte. The electrochemical setup consisted of a conventional three-electrode configuration with a platinum gauze as a counter electrode and a Haber–Luggin capillary with Ag/AgCl (1 M KCl) electrode as a reference electrode. Electrochemical experiments were carried out using high-voltage potentiostat Jaissle IMP 88 in 1 M  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{H}_2\text{SO}_4$ ,  $(\text{NH}_4)\text{H}_2\text{PO}_4$  and  $\text{H}_3\text{PO}_4$  electrolytes with small amounts of fluoride ions at room temperature. All electrolytes

Download English Version:

<https://daneshyari.com/en/article/1472458>

Download Persian Version:

<https://daneshyari.com/article/1472458>

[Daneshyari.com](https://daneshyari.com)