ELECTRONIC SUPPORTING INFORMATION (ESI)

Anodic formation of high aspect ratio, self-ordered Nb_2O_5 nanotubes

Wei Wei, Kiyoung Lee, Santosh Shaw, Patrik Schmuki*

Department of Materials Science, WW4-LKO, University of Erlangen-Nürnberg,

Martensstr. 7, D-91058 Erlangen, Germany

* Corresponding author

E-mail: schmuki@ww.uni-erlangen.de (P. Schmuki)

Experimental Details

For anodization experiments we used niobium foils (0.125 mm thickness, 99.9% purity, Advent). They were degreased by sonicating in ethanol for 10 min, were then rinsed with deionized water (DI) and dried in a nitrogen stream. The electrolytes were prepared from glycerol (99% purity, containing less than 0.5 wt. % of H_2O) mixed with different amounts of NH_4F (0.2 M~0.6 M) and different water additions (0, 5 %, 10 %, 20 %, 50% vol. of DI water). All electrolytes were prepared from reagent grade chemicals (Sigma-Aldrich). All anodization treatments were carried out at room temperature for 2 hours using a three-electrode-system as described previously[1]. We use a platinum flag (2.25 cm^2) at a distance of 2cm from the working electrode as the counter electrode and a platinum wire as the quasi-reference electrode. The potentials reported in the manuscript therefore represent V vs. Pt.

During the anodization process, the voltage was ramped from open circuit potential to the final potential with a sweep rate of 500 mV s^{-1} and then held at the final potential for 2 hours. After anodization, samples were immersed in ethanol for 1 hour to remove remnants of electrolyte and then left to dry in air. Some samples were thermally annealed at 550°C in N_2 atmosphere for 2 hours to crystallize the formed oxide layers.

In order to characterize the morphology of the obtained anodic oxide films, a field-emission scanning microscope (Hitachi FE-SEM S4800) and a transmission electron microscopy (Philips CM 30T/STEM

microscope) were used. Cross-sectional measurements were carried out on mechanically cracked samples. X-ray diffractometry (XRD) was employed for structural characterization of the as-formed and annealed anodic oxide layers at a grazing angle of 1^0, using a X-ray diffractometer (Philips X'pert MPD PW3040 with a Panalytical X'celerator detector) with graphite monochromized Cu Kα radiation (wavelength 1.54056 Å). Characterization of the chemical composition of the formed layers was carried out using X-ray photoelectron spectrometer (XPS, PHI 5600), using Al 2 mm Kα monochromatic radiation (1486.6 eV; 300 W) as the excitation source.

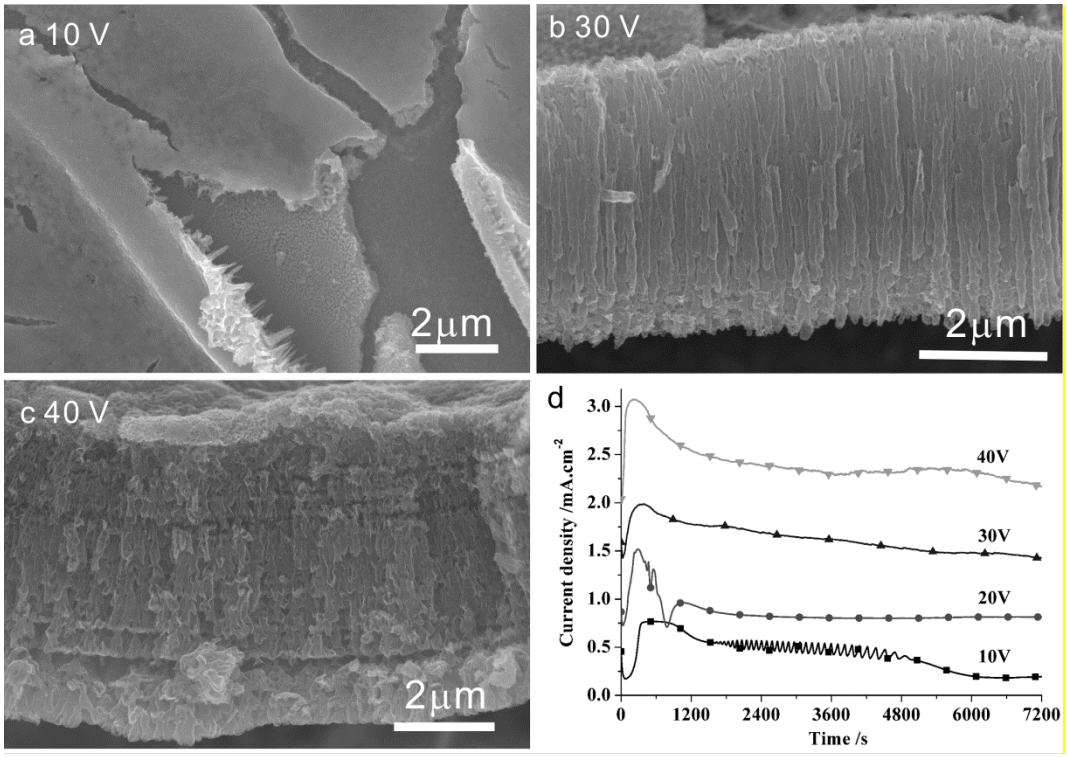

Figure S1 SEM cross-sectional views of the anodic oxide films formed by anodization of Nb in the 0.4M NH_4F containing glycerol electrolyte under different voltages: (a) 10 V, (b) 30 V, (c) 40 V. (d) Current density transients recorded during the anodization of Nb in 0.4M NH_4F containing glycerol electrolytes at different voltages.

Reference

1. J. M. Macak, H. Tsuchiya, L. Taveira, S. Aldabergerova, P. Schmuki, *Angew. Chem. Int. Ed.*, 2005, **44**, 7463.