Synthesis and characterization of SrTiO₃ nanotubes

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Complex metal oxides with the perovskite structure are important materials for various applications in the microelectronics industry and, more recently, in nanotechnology. They can be used as sensors, catalysts and composites with defined electrical properties. For many applications the physical properties of these perovskite ceramics strongly depend on their surface-to-volume ratio. In the case when an increased surface-to-volume ratio is needed this can be achieved by the synthesis of these materials in the form of various nanostructures, such as nanorods and/or nanotubes. Such morphologies exhibit a much increased surface area compared to their bulk counterparts [1, 2]. This is why many efforts have recently been made to synthesize and understand the growth of nanorods and nanotubes of functional ceramics by different processing procedures [3], among which the sol-gel template-assisted method provides a versatile technique for synthesizing one-dimensional nanostructured materials [4].

In our work we report on the synthesis of SrTiO₃ nanostructures by sol-gel electrophoretic deposition (EPD) into template membranes. As the template membranes, commercially available anodic aluminum oxide (AAO) membranes were used, with pore diameters of 200-300 nm and with a thickness up to 60 µm. For EPD of a SrTiO₃ sol into the template, the AAO membrane was attached to an Al working electrode, while a Pt mesh electrode served as a counter electrode. The EPD of the sol was conducted at a potential of 30 V between both electrodes that was maintained for 30 min. After deposition the sample were first dried at 200 °C for 12 h and then annealed at 700 °C for 1 h. The AAO membranes were removed after annealing in 6 M NaOH and the isolated SrTiO₃ nanostructures in the NaOH solution were repeatedly washed in water and ethanol and were concentrated by means of centrifugation.

The obtained SrTiO₃ nanostructures were characterized by scanning and transmission electron microscopy (SEM, TEM). EDXS analysis confirmed that the composition of the observed nanostructures corresponded to stoichiometric SrTiO₃. By using FEG-SEM and CTEM we found that the SrTiO₃ nanostructures grown in AAO templates exhibited tube-like shape (Fig. 1a and 1b), which was additionally confirmed by HAADF-STEM analyses. Figure 1c shows a HAADF-STEM image taken from a part of a single SrTiO₃ tube-like nanostructure. The average intensity profile across the nanostructure was taken from the marked region in the HAADF-STEM image (Fig. 1c). The shape of the intensity profile confirms that the observed SrTiO₃ tube-like shapes are indeed nanotubes. The SrTiO₃ nanotubes grown in AAO membranes are dense, continuous and polycrystalline in nature with a diameter ranging from 200 to 250 nm. The SrTiO₃ grains with a cubic structure are well crystallized with very little or no amorphous phase (Fig 1d). A high-resolution TEM image showed a perfect perovskite pattern with no observable structural defects (Fig. 1d). Figure 2a shows a higher-magnification image of a polycrystalline SrTiO3 nanotube with a grain size distribution in the range from 10 to 25 nm. The comparison between the corresponding selected area electron diffraction (SAED) pattern (Fig. 2b) and the calculated electron diffraction pattern (fig 2c) confirms that the SrTiO3 grains have a cubic structure. Furthermore, single SrTiO₃ crystals with well developed {110} facets exhibit a crystallographic texture, as shown in figure 2d, within the nanotubes. Our results show that the electrophoretic deposition of sols into porous templates is a useful technique for the preparation of one-dimensional SrTiO₃ nanotubes.

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Figure 1. a) TEM and b) FEG-SEM images of an individual SrTiO₃ nanotube grown in an AAO membrane by sol-gel EPD. b) HAADF-STEM image of an individual SrTiO₃ nanotube with the corresponding intensity profile. d) Bright-field TEM image of a single cubic SrTiO₃ grain with the HRTEM image inset showing atom columns in the [001] zone axis.



Figure 2. a) Bright-field TEM image of polycrystalline $SrTiO_3$ nanorod grown in an AAO membrane with the b) corresponding SAED pattern and c) calculated pattern for cubic $SrTiO_3$. d) SAED pattern indicating crystallographic texture of $SrTiO_3$ grains within a nanotube.