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An X-ray and neutron scattering study of self-organized anodic TiO2 nanotubes for battery applications

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Self-organized anodic titania (TiO2) nanotube arrays are an interesting model anode material for use in Li-ion batteries owing to their excellent rate capability, their cycling stability and their enhanced safety compared to graphite. A composite material where carbothermally treated conductive TiO2 nanotubes are used as support for a thin silicon film has been shown to have the additional advantage of high lithium storage capacity. This article presents a detailed comparison of the structure, surface and bulk morphology of self-organized conductive TiO2 nanotube arrays, with and without silicon coating, using a combination of X-ray diffraction, X-ray reflectivity, grazing-incidence small angle X-ray scattering (GISAXS) and time-of-flight grazing-incidence small angle neutron scattering (TOF-GISANS) techniques. X-ray diffraction shows that the nanotubes crystallize in the anatase structure with a preferred (004) orientation. GISAXS and TOF-GISANS are used to study the morphology of the nanotube arrays, delivering values for the inner nanotube radius and intertubular distances with high statistical relevance because of the large probed volume. The analyses reveal the distinct signatures of a prominent lateral correlation of the TiO2 nanotubes of ~94 nm and a nanotube radius of ~46 nm. The porosity averaged over the entire film using TOF-GISANS is 46%. The inner nanotube radius is reduced to half (~23 nm) through the silicon coating, but the prominent lateral structure is preserved. Such in-depth morphological investigations over large sample volumes are useful for the development of more efficient battery electrode morphologies.
Paul N., Brumbarov J., Paul A., Chen Y., Moulin J.-F., Müller-Buschbaum P., Kunze-Liebhäuser J., Gilles R., Journal of Applied Crystallography, 48 (2015) 444 –454.

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