Low-temperature conversion of titanate nanotubes into nitrogen-doped TiO_2 nanoparticles

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Abstract

In the past two decades nanotubes have become the symbol of nanotechnology, a new and fast evolving field of science. Inorganic nanotubes with trititanate structure ($Na_xH_{2-x}Ti_3O_7$) were first synthesized by Kasuga and coworkers. Their intention was to obtain TiO_2 with high specific surface area and enhanced photocatalytic activity. However, the applied alkaline treatment gave rise to morphological changes and tubular nanostructures with diameter of ~8 nm and length of ~100 nm appeared in the samples. Many possible applications of titanate nanotubes are known nowadays: high surface area mesoporous catalyst support, adsorbent, insoluble matrix for ion-exchange processes etc. They are used in the development of lithium-ion batteries, in medical biology and are promising candidates for heterogeneous photocatalysis as well.

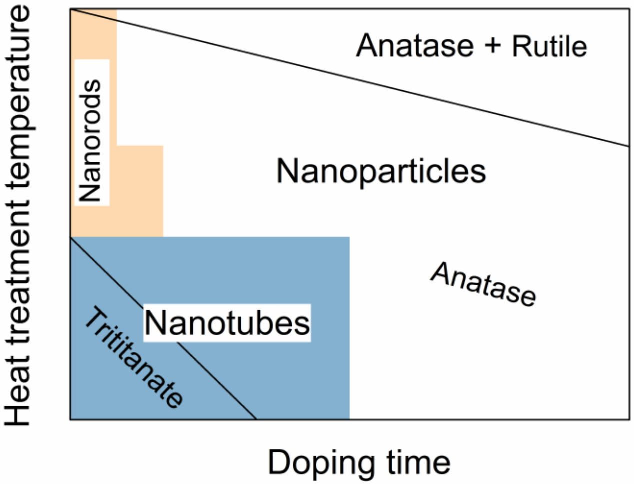
In our study titanate nanotubes were synthesized via the alkaline hydrothermal procedure. After a subsequent protonation step the obtained nanotubes were doped with nitrogen using ammonia formed in situ by the thermal decomposition of urea. This new method is an economic low temperature alternative to the existing gas phase N-doping procedures. Different nitrogen doping times were applied to the samples, followed by calcination steps at various temperatures, while changes in the morphology and phase were investigated. To this end, transmission and scanning electron microscopic (TEM, SEM), energy dispersive X-ray spectroscopic (EDS), X-ray and selective area electron diffraction (XRD, SAED) measurements were performed.

The increase in the nitrogen content and calcination temperature induced changes in the size and shape of the nanotubes, along with the transformation of the crystal structure. Nanotubes were first converted into anatase and then into rutile TiO_2. The initial tubular morphology collapsed, and at low temperatures nanorods, while at higher temperatures nitrogen doped isometric nanoparticles were formed [1].

References

[1] Buchholcz, B.; Haspel, H.; Kukovecz, Á.; Kónya, Z. CrystEngComm, **16** (2014) 7486–7492.

Figures



Morphology and crystalline phase variation of nitrogen-doped titanium-oxide nanostructures with nitrogen doping and calcination temperature.