

Experiment title: Synthesis and transformations of nanomaterials; time resolved in-situ powder diffraction studies.

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CH-2140

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Shifts:	Local contact(s): Yaroslav Filinchuk	<i>Received at ESRF:</i>

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Report:
***In Situ* Studies of Structural Stability of Titanate Nanotubes [1]**

Tao Gao, Helmer Fjellvåg, and Poul Norby

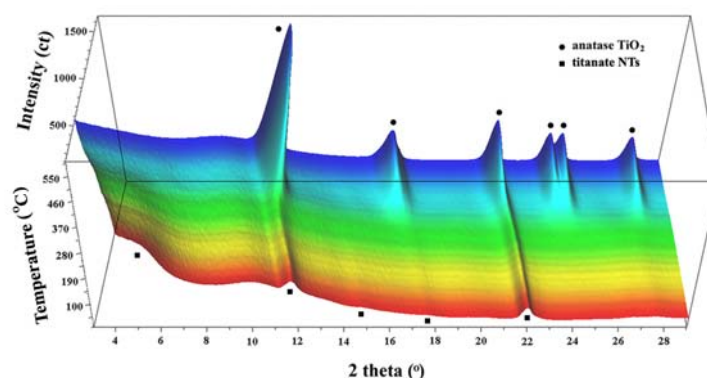


Figure 1. Three-dimensional representation of *in situ* synchrotron XRD data of titanate nanotubes upon heating (wavelength: 0.07106 nm).

Hydrothermally treating crystalline TiO₂ powders with concentrated NaOH solutions results in the formation of titanate nanotubes with high surface area of ~ 400 m²/g. Despite a variety of practical applications ranging from photocatalysts to lithium storage materials have been reported since 1998, microstructures of these nanotubes are still in debate.

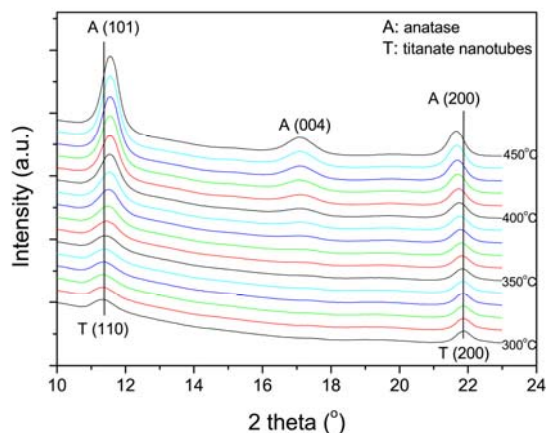


Figure 2. Selected synchrotron XRD patterns near the phase transformation for comparison. The patterns are shifted vertically for clarity (wavelength: 0.07106 nm).

In situ synchrotron X-ray diffraction studies revealed that the titanate nanotubes are thermally unstable at temperatures above 360°C and can transform directly to anatase via a dehydration and recrystallization process (Figure 1, 2). The presence of this direct phase transformation indicated that the titanate nanotubes possess an orthorhombic lepidocrocite (γ -FeOOH)-type layered structure (Figure 3).

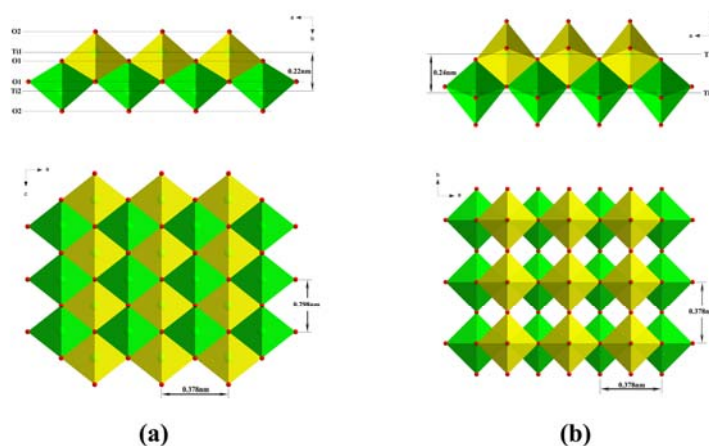


Figure 3. Comparison of lepidocrocite-type titanate layer (a) and the principle layer of anatase (b). The axis notation refers to the original orthorhombic layered titanate and tetragonal anatase.

Reference

- [1] Tao Gao, Harald Fjeld, Helmer Fjellvåg, Truls Norby, and Poul Norby, *In situ studies of structural stability and proton conductivity of titanate nanotubes*, **Energy & Environmental Science**, 2009, DOI: 10.1039/b821532b.