

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.



	Experiment title: The true cascade of structures from oxofluorotitanates with/without presence of V-precursor to VO ₂ TiO ₂ mesocrystal under thermal conversion.	Experiment number: MA4439
Beamline: 28ID (side station).	Date of experiment: from: 24 th October 2018 to: 28 th October 2018	Date of report: 6 th June 2019
Shifts: 12	Local contact(s): Alexei Bosak	<i>Received at ESRF:</i>
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Report:

The experiment was carrying out with instrument on 28ID (side station). We analyzed as-proposed in-situ thermal conversion of NH₄TiOF₃ crystals in presence and without of vanadium precursor into thermochromic mesocrystals VO₂-TiO₂ using both powder and single crystal diffraction. The structure of NH₄TiOF₃ was first decribed nealy 20 yaers ago, as being isostructural with NH₄FeF₄. Our premilinary measurement by WAXS (side station of ID28) indicated that NH₄TiOF₃ is not isostructural with NH₄FeF₄. Detailed study of intermediate phases during the MA4439 accomplished under high resolution at ESRF and favoured to finalized the manuscript on Polar and non-polar structures of NH₄TiOF₃. (J. Appl. Cryst. (2019). 52, 23-26 , <https://doi.org/10.1107/S1600576718016606>)

We described the outcome of single-crystal and powder X-ray diffraction studies, revealing the existence of two polymorphs of the parent NH₄TiOF₃ at different temperatures. A second-order phase transition from the polar Pca21 phase (1), stable at room temperature, to the Pma2 phase (2) above 433 K has been demonstrated. The direction of the pseudo-fourfold axis in NH₄TiOF₃ coincides with the orientation of the fourfold axis of anatase mesocrystals, consistent with a topotactical transformation.

In concerning V-doping, during the experiment time we added up to 60% VO₂ into TiO₂ and controlled transformation via in-situ XRD of NH₄TiOF₃ and V-content precursor (NH₄VO₃) under different atmosphere (air, N₂). Each sample was heated to 650°C using a programmed temperature ramping of 10°C/min and then allowed to cool to room temperature. Extraction of V-doping effects under co-decomposition NH₄TiOF₃ and V-content precursor into VO₂-TiO₂ crystals is in progress.