



**Experiment title:** Tunable synthesis of metastable nanocrystalline phases probed in situ by time resolved XRD and SAXS/WAXS

**Experiment number:**  
**MA-695**

<b>Beamline:</b> ID 11	<b>Date of experiment:</b> from: <b>22 April 2009</b> to: <b>25 April 2009</b>	<b>Date of report:</b> 28 February 2010
<b>Shifts:</b> 12	<b>Local contact(s):</b> Dr G. Vaughan	<i>Received at ESRF:</i>

**Names and affiliations of applicants:**

Dr. Aleksander GURLO (applicant)

Experimentalists: Aleksander Gurlo, Christoph Linck, Ravi Mohan Prasad (Technische Universitaet Darmstadt, Fachbereich Material- und Geowissenschaften, Petersenstr. 23, D-64287 Darmstadt, Germany)

**Abstract:**

The understanding of the transformation mechanism involved in the dehydroxylation reactions in the In-O-H system exhibits large controversy and discrepancy; it holds especially for the formation of the metastable nanosized intermediates as well as for the structural relation between corresponding phases. It was recently reported that indium oxohydroxide (InOOH) appears as an intermediate phase in the thermal dehydroxylation of nanoscaled In(OH)<sub>3</sub>. Our in situ time resolved high energy synchrotron radiation experiments showed unambiguously that no intermediate crystalline or amorphous phases have been observed during the phase transition (dehydroxylation) from nanosized indium hydroxide to indium oxide. Under our experimental conditions, the c-In(OH)<sub>3</sub> to bixbyite-type In<sub>2</sub>O<sub>3</sub> transition was observed between 280 and 305°C and the conversion completed around 305°C without any observable intermediates. The formation of InOOH during the phase transition In(OH)<sub>3</sub> → bixbyite-type In<sub>2</sub>O<sub>3</sub> can be ruled out. This finding is of high relevance and importance for the controllable synthesis of nanocrystalline In<sub>2</sub>O<sub>3</sub>-based materials.

**Publication details:**

L. Schlicker, R Riedel, A. Gurlo, Indium hydroxide to bixbyite-type indium oxide transition probed in-situ by time-resolved synchrotron radiation, *Nanotechnology* 20 (2009) 495702