Evolution and Stability of Ordered Mesoporous Oxides

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Metal oxides are important materials in industry due to their catalytic, optical and magnetic properties. Many of the applications require a high surface area. Oxides with high surface area can be synthesized by introducing a templated porosity into the metal oxide. Typically, employed templates are e.g. amphiphilic block-copolymer. During dip coating, these polymers form micelles that undergo an evaporation-induced self-assembling (EISA) process. Subsequent heat treatments can convert the deposited film into porous oxides via thermal decomposition of the template and crystallization of the framework. However, in many cases the material synthesis can only succeed, when the material precursor is transformed into a structural stable intermediate prior to the decomposition of the templating polymer. Otherwise, the pore structure collapses.

The synthesis of mesoporous magnesium oxide was recently demonstrated using magnesium carbonate as structurally stable intermediate [1]. We investigated if the synthesis procedure can be adapted to produce also mesoporous cobalt oxide. Our preliminary results show that we can synthesize the desired oxide forming a chemical complex with a chelate ligand, where the resulting chemical complex shows the desired stability and decomposition behaviour. For the synthesis a pore template PEO-PB-PEO was mixed with citric acid and Co(NO₃)₂ and dissolved in a mixture of ethanol and water. The films were dipcoated onto Si wafers and thermally treated at different temperatures. SAXS analysis at beamline B1 was used to follow the thermal evolution of the pore structure. To analyse the different directions of the pore system SAXS was measured for different angles between substrate and incident beam.

The recorded 2D-SAXS data is presented in Figure 1. At 90° angle isotropic rings can be observed for all treatment confirming a local nanostructure of the deposited films parallel to the substrate. In contrast, elliptical pattern are observed for 6° analysis indicating pore order also perpendicular to the substrate. However, the films shrink perpendicular to the substrate as seen by the progressing elliptical deformation upon temperature increase. As a result, we were able to show that the recently introduced synthetic strategy can be extended to other metal oxides. The authors are grateful to the HASYLAB for the provision of beamtime under the project I-20110912.

References