Hydrogen is one of the most promising renewable energy sources for mobile and stationary applications for a “green” future. In present applications hydrogen is stored either in liquid or in gaseous form. However, these storage techniques have major drawbacks. Due to those low hydrogen capacities (max. 5wt% hydrogen) and low explosion level of hydrogen, high requirements on the carrier system have to be managed. Hydrogen stored in solid state systems circumvents these problems. However, due to storage capacity, weight and volumetric density of the carrier system, only light metal hydrides are favoured materials for hydrogen storage in mobile applications.

One promising new class of materials are the Reactive Hydride Composites (RHC) [1, 2]. These systems show reduced total reaction enthalpies at high storage capacities. Especially the system of \(2\text{LiBH}_4 + \text{MgH}_2 \leftrightarrow 2\text{LiH} + \text{MgB}_2 + 4\text{H}_2\) shows a few promising features. It has a theoretical storage capacity of 11.4 wt% hydrogen and an equilibrium pressure of 1bar H\(_2\) at 170°C. During the endothermic desorption reaction the exothermic formation of MgB\(_2\) proceeds and thereby lowers the total reaction enthalpy. However, the system shows very slow reaction kinetics and can therefore be presently operated only at temperatures very much above the thermodynamic equilibrium. With suitable catalytic additives the kinetics is improved by an order of magnitude [3]. Characterization of these additives, their chemical state and distribution is the key to understand the hydrogen absorption and desorption mechanisms.

High reaction rates and reversibility are obtained in the 2LiBH\(_4\)-MgH\(_2\) system with addition of NbF\(_5\). To investigate the size and distribution of the Nb phase in the initial as well as in the cycled state, ASAXS measurements at the Nb-K edge were performed.

LiH-MgB\(_2\) composites with 10 mol% NbF\(_5\) additive were prepared by high-energy ball milling. The cycled states were prepared in a Sieverts type titration apparatus. For ASAXS measurements, the powders were enclosed within Kapton tape in the sample holder to avoid oxidation of the samples.

In order to separate the scattering of Nb-containing structures ASAXS measurements at five different energies close to the Niobium K-edge were performed at beamline B1. Additionally XAFS/EXANES measurements were necessary to determine the so-called anomalous dispersions correction factors \(f', f''\) as well the chemical state.

In figure (a), the absorption curves measured in transmission at beamline C at the Nb K-edge at 18986 eV are shown. The RHC samples show a significant shift to lower energies in comparison to the initially added NbF\(_5\) already after milling. A large agreement in edge position as well as in the post edge features are observed with NbB\(_2\). The chemical state upon further cycling appears to be stable. The results are similar to those reported for Zr-based additives [4, 5].
The distance distribution functions of Nb structures in the samples with different cycling states calculated from the pure resonant scattering data using the program Gnom are shown in figure (d). Nanostructures in the range of 11-15 nm are determined. After first desorption larger structures are formed which are stable upon further cycling. Similar results were obtained by investigations of RHC samples with the additives TiF₄ and ScCl₃ performed at low energies at the ASAXS instrument at BESSY/HZB (Berlin).

References