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Deliverable 5.3

Report on upscaling of materials

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Co-ordinator: Maximilian Fichtner, Karlsruhe Institute of Technology, Institute of Nanotechnology

D 5.3 (M 39) Report on upscaling of materials

The content of D 5.3. is similar to D 5.2.

Summary

The wet incipient impregnation and melt infiltration techniques have been established and developed for the preparation of the nanocomposites for the NANOHy project. The melting infiltration has been examined to be a technically possible method to meet the requirement of upscaling the composites. The reversible hydride NaAlH₄ with a melting point at 181 °C is a suitable complex hydride for the melting procedures. Experimental details are similar to those reported in D 3.4 and D 3.5. The essential difference is that more material has been prepared in a bigger autoclave per batch and that several 100 g of the material could be produced successfully in a few batches only.

The characterization of NaAlH₄ nanocomposite has been thoroughly carried out by means of neutron experiments, Raman spectroscopy etc. as reported in D4.2. The investigation of hydrogen storage properties of the nanoconfined NaAlH₄ shows significantly changed thermodynamic and improved kinetic properties as reported in D4.1. Therefore, NaAlH₄ has been chosen as the complex hydride for the optimised nanocomposite for a laboratory test tank of NANOHy. Activated carbon IRH33 prepared by the Institut de recherche sur l'hydrogène (IRH) at the Université du Québec à Trois-Rivières, Canada, has been selected as the support for the preparation of the optimised nanocomposite because of its extraordinarily large micropore volume.

Experimental details

NaAlH₄/carbon nanocomposite

The preparation of the composite has been has been carried out by melting infiltration procedures as described in D3.4.

The as prepared NaAlH₄/AC2 nanocomposite has been characterized by X-ray diffraction measurements. The NaAlH₄ decomposes according to the following reaction:

$$3 \text{ NaAlH}_4 \rightarrow \text{Na}_3\text{AlH}_6 + 2 \text{ Al} + 3 \text{ H}_2 \rightarrow 3 \text{ NaH} + 3 \text{ Al} + 4.5 \text{ H}_2$$

The XRD data of the resulting composites $NaAIH_4/AC2$ is shown in Figure. 1. As a general observation, the diffraction data reveals a partly decomposition of the alanate during the melting procedure.

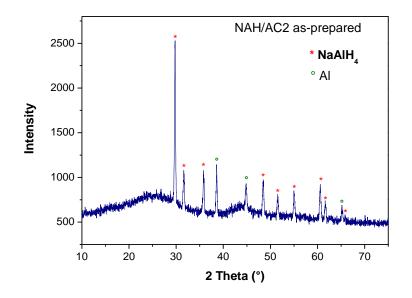


Figure 1. The X-ray diffraction patterns of the NaAlH₄/AC2 composite.

The desorption properties of the NaAlH₄/AC2 composite has been examined by the thermal volumetric measurement with the Sieverts apparatus. Both NaAlH₄/AC2 and NaAlH₄/IRH33 nanocomposite were heated at 150 °C under initial H₂ pressure about 0.2 bar for desorption, then rehydrogenated at 125 °C by applying 115 bar of H₂. The amount of desorbed H₂ has been calculated to weight percent as presented in the Figure 2 and Figure 3. By taking the advantage of the large micropore volume of IRH33, the NaAlH₄/IRH33 nanocomposite shows the higher hydrogen storage capacity than that of NaAlH₄/AC2 composite.

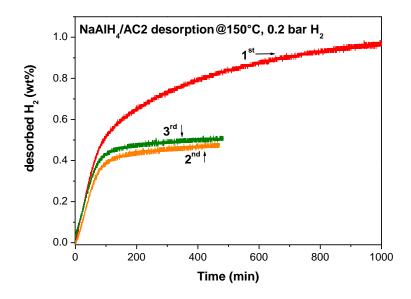


Figure 2. The desorption kinetics of the NaAlH₄/AC2 nanocomposite.

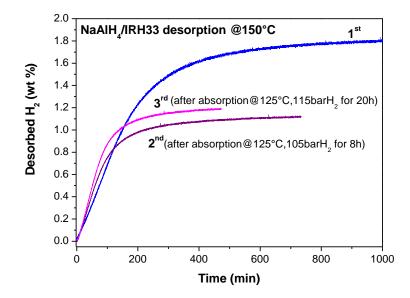


Figure 3. The desorption kinetics of the NaAlH₄/IRH33 nanocomposite.