Electronic Supplementary Material (ESI) for Nanoscale
This journal is © The Royal Society of Chemistry 2013

Nanoconfined $NaAlH_4$; Prolific Effects from Increased Surface area and Pore Volume

Thomas K. Nielsen,[1] Payam Javadian,[1] Marek Polanski,[2] Flemming Besenbacher,[3]
Jerzy Bystrzycki,[2] Jørgen Skibsted,[4] Torben R. Jensen[1]*

[1] Center for Energy Materials, Interdisciplinary Nanoscience Center (iNANO), and Department of Chemistry, Aarhus University, DK-8000 Aarhus, Denmark

[2] Faculty of Advanced Technology and Chemistry, Military University of Technology, 2 Kaliskiego Str., 00-908 Warsaw, Poland

[3] Interdisciplinary Nanoscience Center (iNANO) and Department of Physics and Astronomy, Aarhus University, DK-8000 Aarhus C, Denmark

[4] Instrument Centre for Solid-State NMR Spectroscopy, Department of Chemistry, and Interdisciplinary Nanoscience Center (iNANO), Aarhus University, DK-8000 Aarhus C, Denmark

* Corresponding author: (T. R. Jensen)

*Corresponding author
Torben R. Jensen, Ph.D., Associate Professor
Center for Materials Crystallography
iNANO and Department of Chemistry
Langelandsgade 140
DK-8000 Aarhus C
Aarhus University
Denmark

E-mail: trj@chem.au.dk

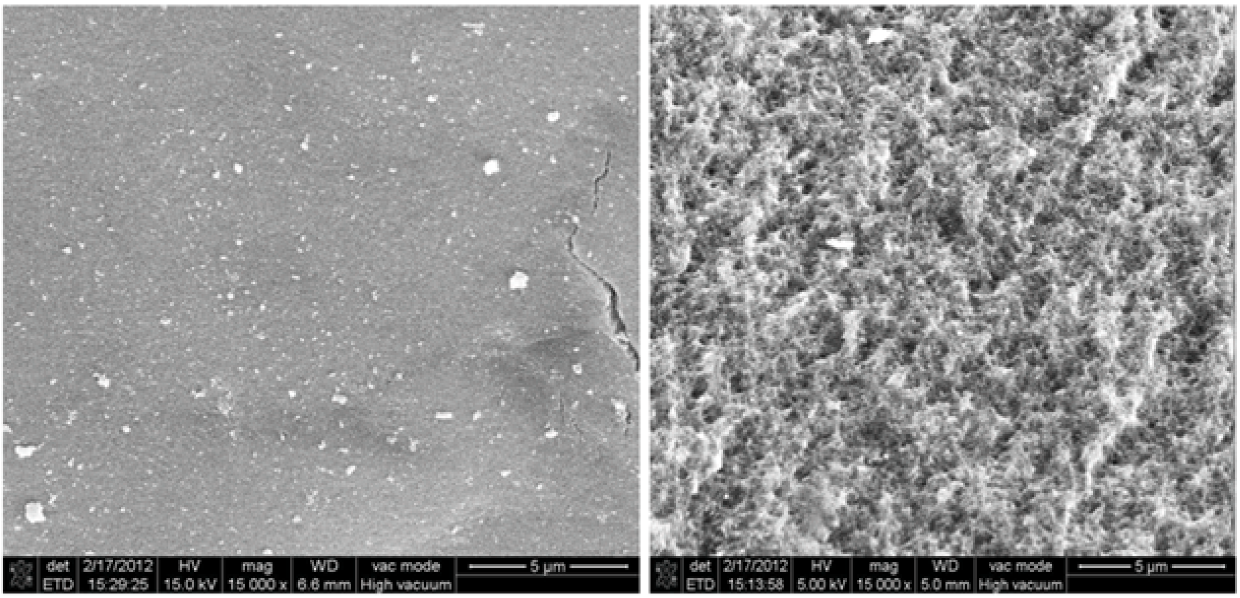

Figure S1. SEM images of as prepared resorcinol formaldehyde carbon aerogel, D_{max} = 10 nm (left), denoted CA, and CO_2 activated aerogel (950 °C in a CO_2 flow for 5.1 hours) denoted CA-5.1, after infiltration of $NaAlH_4$. The micrometer scale is chosen to better illustrate the significant differences in morphology.

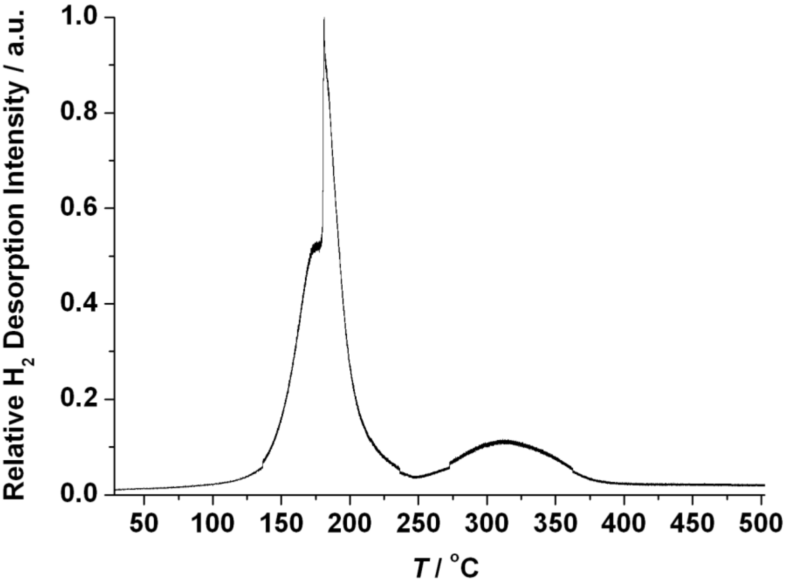

Figure. S2. Normalized TPD-MS data showing the relative hydrogen desorption intensities for $NaAlH_4$ infiltrated in CA-5 recording the mass spectrometer intensity of H_2^+ ions (m/e = 2) collected in the temperature range R*T* to 400 °C ($\Delta T/\Delta t$ = 1 °C/min).

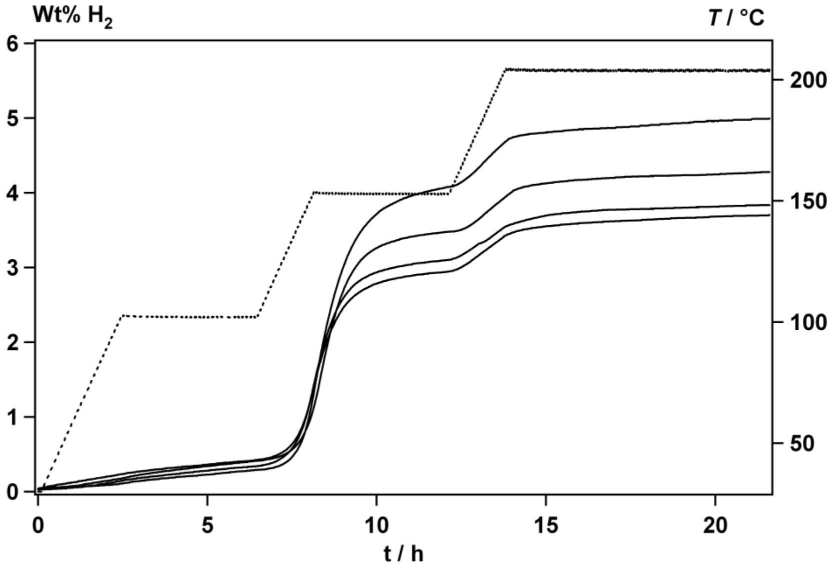

Figure S3. Sieverts' measurements showing hydrogen desorption cycles 1 to 4 for $NaAlH_4$ nanoconfined in CA-5. Hydrogen desorption was performed at fixed temperatures of 100, 150, and 200 °C ($\Delta T/\Delta t$ = 0.5 °C/min; dashed lines). Hydrogen absorption was performed at 160 °C and $p(H_2)$ = 90 to 93 bar during 10 hours.

CO_2 - activated aerogels.

A resorcinol formaldehyde carbon aerogel, RF-CA, with a pore size, D_{max}, for which the pore size distribution reach a maximum value of 10 nm was selected for this study and denoted CA. The morphology of RF-CA's can be systematically designed by activation in CO_2.[1-2] Sample CA was heated in a stream of CO_2 in different periods of time and the specific surface area, the micro porosity and total pore volume gradually increased while the pore size, D_{max}, remained less affected, in accordance with previous studies.[1-2] The obtained aerogel morphology may on the crucible position in the tube furnace, i.e. the temperature gradient in the furnace. This may contribute to the significant difference in surface area for samples CA-5 and CA-5.1 both activated during ca. five hours. Therefore, also aerogel monoliths from the same batch may have experienced different heating profiles depending on their individual positions in the 10 cm long crucible and maybe also different gas flow. The texture parameters given in the manuscript, Table 1, are estimated based on this assumption.

Table S1. Texture parameters for the as prepared carbon aerogel (CA) and activated scaffolds by heating in a flow of CO_2 and $TiCl_3$-functionalized (CA-Ti) carbon aerogel (CA) scaffold materials.

Sample	CO_2 - time (h)	S_{BET} (m^2/g)	V_{meso} (mL/g)	V_{tot} (mL/g)	V_{micro}/V_{tot}	D_{max} (nm)
CA	0	704	0.74	0.91	0.19	10
CA-3	3	1267	0.96	1.30	0.28	11
CA-5	5	1977	1.38	1.94	0.29	10
CA-5.1	5.1	1966-2505	1.27-1.58	1.84-2.38	0.29	8
CA-Ti [a]	5.1	1795-2287	1.16-1.44	1.68- 2.17	0.27	8

a) The $TiCl_3$ content was 8.7 wt%

[1] Lin, C.; Ritter, J. A. *Carbon* **2000,** *38*, 849-861.
[2] Al-Muhtaseb, S. A.; Ritter, J. A. *Adv. Mater.* **2003,** *15*, 101-114.
[3] Nielsen, T. K.; Polanski, M.; Zasada, D.; Javadian, P.; Besenbacher, F.; Bystrzycki, J.; Skibsted, J.; Jensen, T. R. *ACS Nano* **2011,** *5*, 4056-4064.