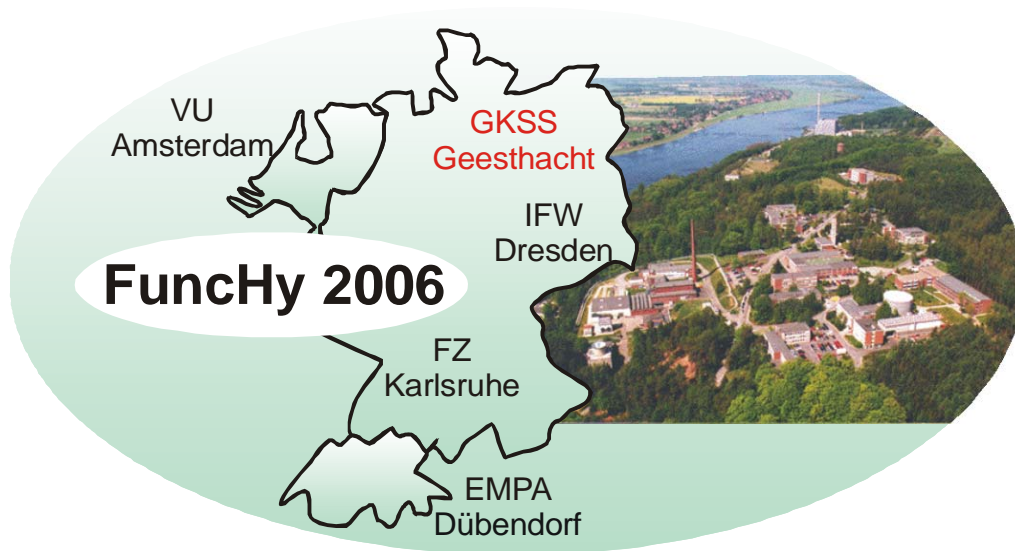


FuncHy 2006

1st Workshop of the Helmholtz Initiative

Functional Materials for mobile Hydrogen Storage



GKSS Research Centre Geesthacht,

September 20-22, 2006

Locations

Accommodation:



RAMADA Hotel Hamburg-Bergedorf, Germany

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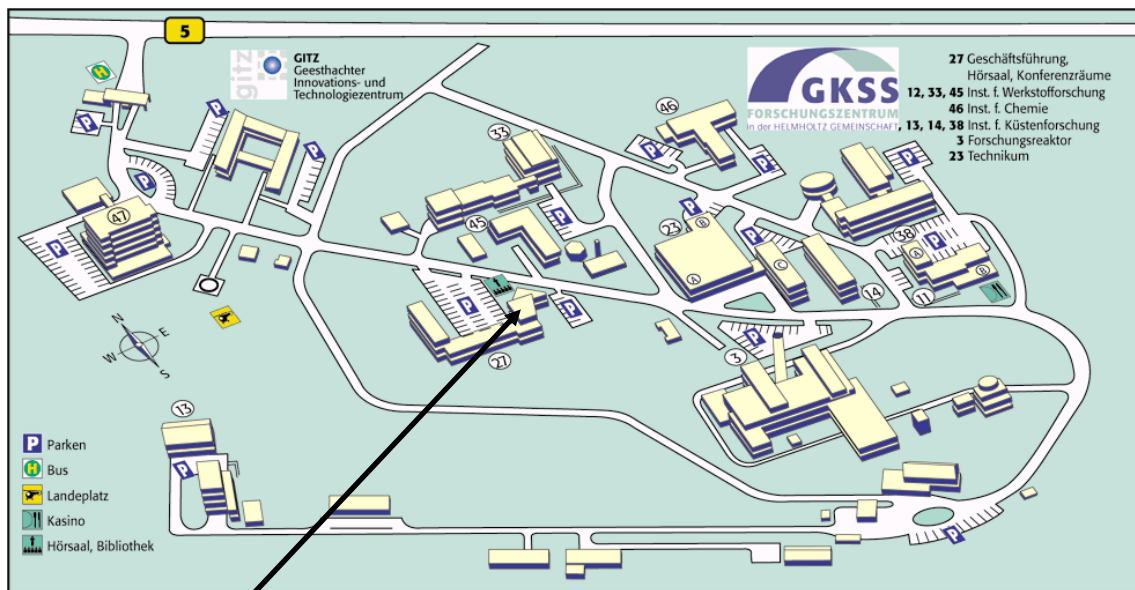
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Workshop:

**GKSS Research Centre Geesthacht GmbH, Institute for Materials Research,
Max-Planck-Straße 1, D-21502 Geesthacht, Germany**

<http://www.gkss.de>



Lecture hall: building 27 ('Hörsaal, Bibliothek')

Secretary: Sabine Schrader

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Time Schedule and Overview of the Workshop

| 20.09.06 | 21.09.06 | 22.09.06 |
|---|---|---|
| 8:30 | 8:30 Bus transfer to GKSS | 8:30 Bus transfer to GKSS |
| 9:15 | Session Chair: Bernard Dam, VU 9:15 R. Bormann, GKSS: Welcome and Introduction 9:30 M. Felderhoff, MPI-Mühlheim: Doped NaAlH₄ – Catalytic and Thermodynamic Effects 10:05 M. Fichtner, FZ Karlsruhe: Properties and Transformation of Alanates <hr/> 10:40 Coffee break Session Chair: Maximilian Fichtner, FZK 10:55 N. Eigen, GKSS: Integrating Complex Hydrides into Hydrogen Storage Systems 11:30 A. Züttel, EMPA Zürich: Properties of Boronates 12:05 M. Dornheim, GKSS: Mg-based Reactive Hydride Composites for Hydrogen Storage? | Session Chair: Thomas Klassen, HSU 9:15 O. Løvvik, Uni Oslo: First Principles Modelling for Predicting and Understanding novel Complex Hydrides. 9:50 B. Dam, E. Batyrev VU Amsterdam: Determination of Thermodynamic Properties of Gradient Films using Hydrogenography 10:25 S. Doppiu, IFW Dresden Determination of Thermodynamic Properties of Complex Hydrides <hr/> 11:00 Coffee break Session Chair: Oliver Gutfleisch, IWF 11:15 W. Lohstroh, FZ Karlsruhe: Nitride based Hydrogen Storage Materials: The Li-Mg-NH System 11:50 M. Hirscher, MPI Stuttgart: Hydrogen Storage by Physisorption in Microporous Materials |
| | 12:45 Lunch (Foyer Lecture Hall) | ca. 12:30 End of the Workshop |
| | Session Chair: Andreas Züttel, EMPA 14:00 E. Schmidt-Ihn, DaimlerChrysler: Requirements and Expectations for an On-board Hydrogen Storage System 14:35 U. Eberle, GM Fuel Cell Activities: GM's Research Strategy towards a Hydrogen-based Propulsion System <hr/> 15:10 Coffee break Session Chair: Jobst Hapke, TUHH 15:25 Michael Stoecklin, BMW: Overview of Hydrogen Storage Activities at BMW 16:00 Michael Kahlich, VW AG: Automotive Requirements for Future Hydrogen Storage Systems <hr/> 17:00 Discussion Recent results, opportunities and challenges <hr/> 18:30 Bus transfer to Hotel Buffet | 13:00 Bus transfer to Hamburg Main Station and Hamburg Airport Opportunity for Visit to Institute for Materials Research |
| 19:00 Get together at the Ramada-Treff Hotel | 19:30 Italian Buffet (Ramada-Treff Hotel) | |

Welcome and Introduction to the FuncHy Project

Rüdiger Bormann

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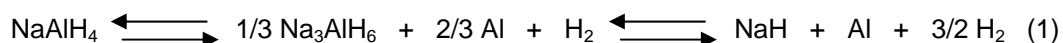
Exploratory focus of the Helmholtz-Initiative 'FuncHy: Functional Materials for mobile Hydrogen Storage' is the development of novel light-weight hydrogen storage materials with high gravimetric capacity (> 5 mass %), low operating temperatures (< 150 deg C) and fast sorption kinetics. The project combines various scientific and technical approaches and methods, considering the total value-added chain from fundamentals of materials science to production on industrial scale. For this, FuncHy assembles five renowned European research institutions with complementary experience in the field of hydrogen storage materials. Together with 13 associated partners from industry and academia, FuncHy provides a basis for a European centre of excellence for hydrogen technology for mobile applications.

Doped NaAlH₄ – Catalytic and Thermodynamic Effects

Michael Felderhoff

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One of the main goals for the success of zero emission cars driven by fuel cells is the simple, safe and low cost storage of pure hydrogen. The presently widely investigated sodiumaluminiumhydride (NaAlH₄), with a theoretical hydrogen storage capacity of 5.6 wt.-% offer an alternative solution to high pressure or low temperature hydrogen storage methods. The reversible dissociation process of NaAlH₄ takes place in a two step mechanism via Na₃AlH₆ as an intermediate under Ti-catalysis as shown in Eq. 1.



Our investigations were directed towards better understanding of the catalyst system and accelerating the de- and rehydrogenation rate of doped NaAlH₄. We therefore searched for new catalysts and a fast and simple doping procedure for this material. Thermodynamic measurements and isotope-scrambling experiments provide an insight to the understanding of the doped NaAlH₄ system.

Properties and Transformation of Alanates

Maximilian Fichtner, Aline Leon, and Christoph Frommen

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Mechanical and chemical synthesis methods have been applied for the preparation of ternary and quaternary complex aluminum hydrides. The peculiarities of both methods will be presented as well as the state of the art with properties that are relevant for reversible Hydrogen storage.

Moreover, the transformation mechanism of alanates has been elucidated by various methods and a suggestion is made for the fate and the role of Ti dopants in the transformation of the Al-based storage materials.

Integrating Complex Hydrides into Hydrogen Storage Systems

Nico Eigen, Claude Keller, Martin Dornheim, Thomas Klassen, Rüdiger Bormann

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The integration of complex hydrides into a tank system necessitates the investigation and optimisation of the hydrogenation behaviour of larger amounts of these materials. For room temperature hydrides, hydrogen storage systems up to several kilograms of hydrogen have demonstrated to be feasible, absorbing and desorbing the hydrogen in a few minutes. The knowledge of hydrogenation behaviour and heat transfer in these materials allows for simulation and layout of the tank system based on critical material data. However, in complex hydrides, the phase transformations and the severe morphology and volume change occurring during hydrogenation as well as a lack of data on heat transfer are challenging for the tank designer.

A suitable approach to the complex problem of system design and production of materials is presented. The influence of different methods of material production on the hydrogenation behaviour is addressed. In particular, the limiting steps for hydrogenation with a focus on heat and mass transfer mechanisms are discussed in order to find feasible solutions for the design of hydrogen storage systems based on complex hydrides with high gravimetric storage capacity and high sorption kinetics.

Properties of Boranates $M[BH_4]_x$

Andreas Züttel

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One of the most interesting features of the metallic hydrides is the very high volumetric density of the hydrogen atoms present in the host lattice [1]. The greatest volumetric hydrogen density known today [2] is $150 \text{ kg}\cdot\text{m}^{-3}$ found in Mg_2FeH_6 and $Al(BH_4)_3$, both hydrides belonging to the complex hydrides. The structure of p-element complex hydrides is different from the structure of the consisting metallic elements. The hydrogen in the complex hydrides is often located in the corners of a tetrahedron with boron or aluminum in the center. The negative charge of the anion, $[BH_4]^-$ and $[AlH_4]^-$ is compensated by a cation e.g. Li or Na. The electron from the cation is to a large part localized on the boron or aluminum leading to a mainly ionic bond between the cation and the anion. Within the $[BH_4]^-$ and $[AlH_4]^-$ tetrahedrons the bonds are mainly covalent. While the alanates do not form any volatile AlH_x compounds the boranates desorb beside the hydrogen sometimes also B_xH_y compounds. Therefore it is essential for the hydrogen desorption to break the covalent bonds and not preliminary the ionic for hydrogen desorption from the boranates.

The structure and sorption properties of $Li[BH_4]$, $Na[BH_4]$ and $K[BH_4]$ were investigated. Furthermore, $Mg[BH_4]$ was synthesized from $Li[BH_4]$ is reacted with $MgCl_2$ by thermal activation under hydrogen pressure. The product desorbs hydrogen at significantly lower temperature as compared to $Li[BH_4]$.

- [1] Züttel A, "Hydrogen Storage Methods", Naturwissenschaften 91 (2003), pp. 157-172
- [2] L. Schlapbach and A. Züttel, „Hydrogen-storage materials for mobile applications”, NATURE 414 (15 Nov. 2001), pp. 353-358

Mg-based Reactive Hydride Composites for Hydrogen Storage?

**Martin Dornheim, Gagik Barkhordarian, Ulrike Bösenberg, Andreas Borgschulte,
Nico Eigen, Xiumei Qi, Claude Keller, Oliver Metz, Thomas Klassen, Rüdiger Bormann**

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So far metal hydrides have not been considered competitive for hydrogen storage in most mobile applications because of a low gravimetric hydrogen storage density or a necessary and in many cases very complex heat management system. However, recent developments in this area give reason to the conviction that this could change in the near future.

The preparation of nanocrystalline hydrides using high-energy ball milling as well as the discovery of suitable catalysts/dopants enabled the production of novel lightweight materials for hydrogen storage with much higher gravimetric hydrogen storage densities and fast ab- and desorption kinetics. Hydrogen absorption and desorption in catalyzed lightweight hydrides like MgH_2 and NaAlH_4 now is possible within a few minutes. However, the bonding enthalpies of these new storage materials are still too high or the gravimetric storage density is still too low.

A very exciting and promising novel approach is the concept of the Reactive Hydride Composites (RHC). Such systems show very high gravimetric hydrogen storage densities and reduced total reaction enthalpies as well as significantly improved absorption kinetics compared to the pure hydrides. In Mg-based RHCs reversibility is demonstrated for several borohydrides.

In this presentation results of the sorption behaviour of such new Mg-based composites are shown and discussed.

Requirements and Expectations for an On-board Hydrogen Storage System

Eberhard Schmidt-Ihn, Josef Zieger

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The on-board hydrogen storage is one of the bottle-necks for the implementation of a hydrogen based mobility. In the lecture a short overview about the hydrogen history at DaimlerChrysler is given and the state of the art of on-board storage systems is shown. The requirements for a hydrogen on-board storage system are derived from the data of a present Compact Car. The goals of the DOE and the EU sponsored StorHy project are shown. The expectations for a storage system from the automotive point of view and the main focus for the future development at DaimlerChrysler are discussed.

GM's Research Strategy towards a Hydrogen-based Propulsion System

Ulrich Eberle

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Within the framework of this talk, the research strategy of General Motors on hydrogen storage tank systems shall be shortly presented. A special focus will be the technology status and research needs for systems based on solid-state materials. However, a major technology improvement is still needed for the on-board fuel storage system since hydrogen exhibits a rather low volumetric energy density compared to conventional fuels like diesel or gasoline. Furthermore, the cost for current hydrogen containers is far from those of conventional tanks. Therefore, alternatives (e.g. metal hydrides, complex hydrides or high-surface materials) are investigated for their feasibility within the framework of GM's "Hydrogen Storage Innovation Program". These kinds of systems show very high volumetric storage densities on a materials basis. Novel results of this program (and those of our research partners) will be briefly described. Unfortunately, these hydrogen containers are usually quite heavy resp. bulky and thus possess a low gravimetric resp. volumetric energy density on a systems base. Especially the fundamental thermodynamics and kinetics of the absorption/desorption reactions cause an important engineering burden. The most promising hydrogen storage technologies and their advantages and drawbacks will be described and compared to those of competing fuels. Eventually, promising research routes shall be discussed.

Overview of Hydrogen Storage Activities at BMW

Michael Stoecklin, Jürgen von Wild

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From 1979 until 2001, five generations of hydrogen powered cars had already been demonstrated. Throughout these decades of research and development, the BMW Group has become the world's leader in the practical use of hydrogen. BMW is therefore clearly committed to the use of hydrogen in the combustion engine offering the widest range of benefits due to its dual-mode operation, its dynamic performance and high degree of all-round maturity. A version of the current BMW 7 Series, the 6th generation hydrogen vehicle, is already going through the process of series development.

The presentation will show the six generations of hydrogen powered cars with examples of requirements and the resulting challenges, which must be tackled during the development process at BMW. After that, some examples of international projects and cooperation with involvement of BMW will be given.

Another focus of the talk will be on novel liquid hydrogen storage tank systems. As an example, the Subproject "Cryo" of the European Union funded "StorHy" Project will be shown. The objective of Subproject Cryo is the design of car structure adapted liquid hydrogen lightweight tank system, called "HylighT". New results of that research project will be presented.

Automotive Requirements for Future Hydrogen Storage Systems

Michael Kahlich

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Energy density and cost structure of conventional hydrogen storage devices remain an issue to the marketability of future drive train concepts. Initially the different scenarios for meeting the customer expectations on vehicle range and system costs are shown. Starting from the current status of automotive hydrogen storage the complete system approach to alternative concepts is presented. Derived from this approach the constraints of the automotive application are addressed and transferred into the general specifications of a solid state hydrogen storage system. These are further explained from the technical perspective of the vehicle balance of plant and the potential to interact with the hydrogen storage system. Finally the main topics for a baseline material research to meet the goals of mobile applications are summarized

First Principles Modelling for Predicting and Understanding novel Complex Hydrides

Ole Martin Løvvik

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Atomistic modelling from first principles is an increasingly important tool to aid and complement experimental studies of materials. It is in this talk demonstrated how this may be accomplished for three different cases involving complex metal hydrides relevant for hydrogen storage. It is first shown how a systematic search for unknown compounds may be performed by using a number of input structure models. This is applied to mixed-alkali alanates, e.g. mixtures of NaAlH_4 and LiAlH_4 . This led to the prediction of some previously unknown compounds, which now have been confirmed experimentally. One such mixed alanate is a mixture of Na_3AlH_6 and Li_3AlH_6 : $\text{Na}_2\text{LiAlH}_6$. This compound has been studied by a number of experimental studies, but its thermodynamic phase diagrams have largely been unknown. It is shown how the combination of first principles DFT calculations, phonon calculations and thermodynamic modelling gives results that are consistent with experimental data and point to the maximal theoretical hydrogen reversibility within the Na-Li-Al-H system. Finally, modelling results on Ti-enhanced NaAlH_4 is presented. Large experimental efforts have been carried out to understand the effects of Ti on the hydrogenation properties of NaAlH_4 , but our understanding is still quite limited. This is most probably because the Ti majority phase is not the active one, which hinders the direct experimental observation of the most interesting Ti-containing phase. Modelling efforts have now identified not only possible minority phases containing Ti, but also suggested possible mechanisms that may explain the improved hydrogenation kinetics of NaAlH_4 when Ti is added.

Determination of Thermodynamic Properties of Gradient Films using Hydrogenography

Bernard Dam, R. Gremaud, Erdni Batyrev, D. Borsa, Ronald Griessen,

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Hydrogenography is a novel technique that exploits the optical changes induced by hydrogen absorption in metal thin films. In this contribution, we will show how we measure the kinetic and thermodynamic parameters of a given hydrogen-metal system.

As a model system, we use $\text{Mg}_x\text{TM}_{1-x}$ thin films with a compositional gradient produced by co-sputtering from off-centred magnetron sources. By gradually increasing the pressure and measuring the associated change in optical transmission, we determine the equilibrium pressures of all compositions x in a single run. Indeed, we find that the thermodynamics of hydrogenation depends on the composition. From measurements at various temperatures, the enthalpy of formation of each individual composition x is obtained.

A promising system for application in batteries and solar collectors is the Mg-Ti-H system. Using hydrogenography we determine the kinetics and thermodynamics of the $\text{Mg}_x\text{Ti}_{1-x}\text{H}_y$ phases including the fluorite-type hydride phase which appears to be stable and reversibly formed in our thin films.

Presently, we are setting-up a deposition system for in-situ hydrogenography, to determine the thermodynamics of complex metal hydrides such as NaAlH_4 , which is extremely oxygen sensitive. The UHV deposition system is also connected with an UHV-STM for in-situ surface analysis of the hydride and its catalyst. The surface characterisation analysis includes STM in combination with STS mapping and AFM to study surface modifications of post-loaded films. Thermal desorption spectroscopy will be performed in attached preparation chamber to determine the hydrogen desorption behaviour from hydrogenated films. Again a combinatorial approach will be applied for a search of new catalysts.

Determination of Thermodynamic Properties of Complex Hydrides

Stefania Doppiu, Ludwig Schultz, Oliver Gutfleisch

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The evaluation of the thermodynamic properties and the mechanism involved during the decomposition of complex hydrides is a key issue in order to design new synthesis routes to obtain materials with improved properties for hydrogen storage application. The hydrogen release of these compounds is characterized by a sequence of transformations often including the melting of the hydride. The understanding of the mechanism involved in the decomposition/recombination reaction of this compounds as well as the effect of modification induced by catalysts addition appear fundamental to improve stability and kinetic behaviour.

In the present study alanates, borohydrides and reactive hydride composites will be presented.

The combination of different techniques, such as differential scanning calorimetry under hydrogen pressure, thermogravimetry, volumetric measurements and ball milling under hydrogen pressure, for the characterization of these materials will be discussed.

Nitride based Hydrogen Storage Materials: The Li-Mg-NH System

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Since the discovery of Chen et al. [1] that mixtures of LiNH_2 and LiH can reversibly store 10.5 wt. % H_2 , amide based systems are considered promising candidates as storage materials in a future hydrogen economy. Half substitution of Li by Mg lowers the decomposition temperature and it was found that the Li-Mg-NH system can deliver 300 bar H_2 at 200 °C. The reversible hydrogenation reaction takes place between $\text{Mg}(\text{NH}_2)_2 + 2\text{LiH}$ in the absorbed state and $\text{Li}_2\text{Mg}(\text{NH})_2$ in the desorbed state [2]. One major obstacle for using nitride based systems is the potential release of volatile ammonia. We will present results on the reaction steps and ammonia release in Li-Mg-NH with various added catalysts. Ti-based additives lower the desorption temperature of the initial 2LiNH_2 - MgH_2 mixture while the addition of Ti-based additives after the preparation of $\text{Mg}(\text{NH}_2)_2 + 2\text{LiH}$ had only negligible influence on both the desorption temperature and the amount of released ammonia.

[1] P. Chen, ZT. Xiong et al., Nature 420 (2002) 302

[2] W. Luo and S. Sickafoose, J. Alloys Comp. 407 (2006) 274

Hydrogen Storage by Physisorption in Microporous Materials

Michael Hirscher, Barbara Panella

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One promising possibility to reversibly store hydrogen is adsorption of hydrogen molecules on novel nanoscale materials.

Carbon nanomaterials show interesting properties, e.g. high specific surface area, microporosity and low mass. Apart from these characteristics, materials like activated carbons are cheap and easy to produce. Zeolites are aluminosilicates with a very open microporous structure with channels that are large enough to contain gas molecules. Moreover, the presence of electrostatic forces within the channels is a unique characteristic of the Zeolites. In recent years, a new class of materials with high specific surface area, the metal-organic frameworks (MOFs) have been developed and studied for gas adsorption. These MOFs consist of metal oxide clusters connected by rigid aromatic linkers giving a three dimensional porous structure and, furthermore, they possess the lowest density of all known crystalline materials.

The presentation will give an overview of the experimental situation of hydrogen storage media based on physisorption comparing carbon materials, Zeolites and MOFs. The results indicate that nanostructures with high specific surface area and micropore density are very promising materials for hydrogen storage by physisorption at lower temperatures.

Helmholtz Initiative FunchHy **Functional Materials for Mobile Hydrogen Storage**

Core-partners:

GKSS Research Centre Geesthacht GmbH, Institute for Materials Research,
Prof. Dr. R. Bormann (co-ordinator)

Forschungszentrum Karlsruhe GmbH, Institute for Nanotechnology,
Prof. Dr. H. Hahn

Leibniz Institute for Solid State and Materials Research Dresden, Institute for Metallic Materials (IMW),
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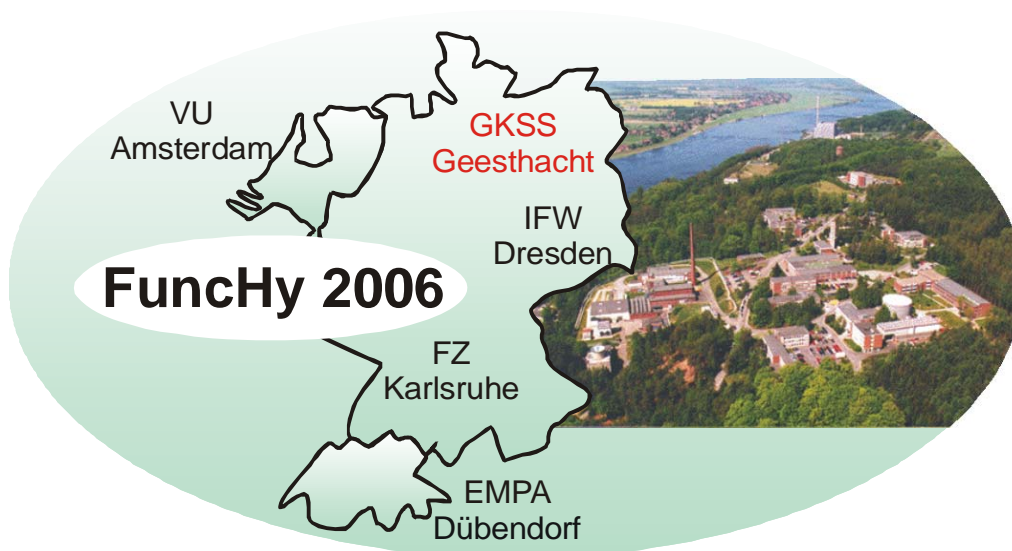
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How to reach GKSS

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The GKSS Research Center is located about 30 km southeast of Hamburg.
To get to us:

from the airport Hamburg-Fuhlsbüttel to Hamburg Hauptbahnhof

- ⇐ by airport shuttle bus to main railway station Hamburg Hauptbahnhof (30 minutes), departure every 30 minutes

from main railway station Hamburg Hauptbahnhof

- ⇐ by suburban railway S2 or S21 to Hamburg-Bergedorf (20 minutes)
- ⇐ there take bus No. 31 or 231 to Geesthacht Markt up to bus stop "Grüner Jäger" (30 minutes), then approx. 20 minutes by feet
- ⇐ or take bus METRO 12 up to „Geesthacht ZOB“ (20 minutes) and then take a taxi to GKSS (10 minutes)
- ⇐ or just inform us in advance so that we can arrange a pick-up for you from suburban railway station „Hamburg-Nettelburg“

by car:

- ➔ from Berlin on A 24 use exit "Büchen", follow the road to Lauenburg, then the B 5 to Geesthacht
- ➔ from the south follow the A 1 to Hamburg, then direction Lübeck, then use the A 25 to Geesthacht and the B 5 to Lauenburg. The GKSS is located on right about 3 km after Geesthacht

by taxi

- ⇐ from the airport approx. 60 minutes / EUR 60,--
- ⇐ from railway station Hamburg Hauptbahnhof approx 45 minutes / EUR 40,--
- ⇐ from Geesthacht „Marktplatz“ approx. 10 minutes / EUR 8,--

Traffic information:

<http://www.hvv.de>

Registration fee

The registration fee is €150,-

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