Detailed Description of Improved Research Facilities

H2FC

EUROPEAN RESEARCH INFRASTRUCTURE

Transnational Access @ KIT

Description of the infrastructure
Name of the infrastructure: HYKA, MHtest, SOFCTEST
Location (town, country): Karlsruhe and Eggenstein-Leopoldshafen, Germany
Web site addresses: www.kit.edu , www.iket.fzk.de, www.iwe.kit.edu
Legal name of organisation operating the infrastructure: Karlsruhe Institute of Technology (KIT)
Location of organisation (town, country): Karlsruhe, Germany
Annual operating costs (excl. investment costs) of the infrastructure (€): 440.000 € (as of 2009)

1. Running Transnational Access Activities

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2006: MgB2-based Superconducting Magnetic Energy Storage - Investigation of H2-Related Aging Effects & Compatibilities

Background: Due to an increase of the intermittent contribution of renewable power plants (e.g. wind/solar) to the public electricity grid an increasing need for balancing demands and supplies is anticipated. Liquid Hydrogen (LH2) is one prime candidate for large scale stationary energy storage, but balancing load/supply fluctuations with hydrogen alone is unrealistic. So the novel hybrid energy storage concept LIQHYSMES for variable renewable energy combines the use of LIQuid HYdrogen for large scale longer-term stationary storage with Superconducting Magnetic Energy Storage (SMES) to provide high power over short time scales like seconds or minutes. The SMES on the basis of the new MgB2 superconductor can be operated in a LH2 bath which allows the cryogenic infrastructure of the LH2 storage also to be used for the SMES part of the storage system. Due to this synergic effect losses and costs are significantly reduced. But the superconducting parts and electrical elements of the SMES have to be qualified concerning their compatibility with LH2 since aging effects due to H2 diffusivities or embrittlement have to be excluded for the anticipated long-term operation of the SMES. So selected parts and components of the SMES were exposed directly to LH2 at KIT-HyKA to investigate their compatibility with LH2.

Results: The parts to be tested were delivered by the user after undergoing pre-test analysis in the labs at Columbus Superconductors spa. The test rig comprised of two large super-insulated vessels for LH2 and one LN2 tank. The first LH2 vessel, a cryostat vessel, had a large removable lid and was used for the experiments in which the specimens were exposed to LH2 while the second one was a storage vessel for additional LH2, since during the experiments large quantities of LH2 were lost due to evaporation. LH2 has a boiling temperature of approx. 21 K and is quite expensive, so LN2 was used as cooling fluid in the outer shell of the double insulated experimental vessel for the first cooling step of vessel and specimens to a temperature of approx. 77 K at the beginning of the experiment, but also later to minimize the LH2 losses due to heat transfer from the outside. After the pre-cooled specimens were exposed to LH2 the temperature in the experimental vessel was monitored continuously and from time to time either LH2 had to be added to the experimental vessel or LN2 to the cooling circuit of this vessel. The specimens were exposed to LH2 for 3 ½ days before the vessel was allowed to warm up slowly. During this warm-up phase the specimens were exposed to the whole temperature range from 20 K to room temperature, since this was also found to be necessary for some test samples. After the experiment the specimens were returned to the user for the post-test analysis in their labs (KIT-HyKA offers no analysis installations in the frame of the H2FC-Transnational Access). In total the facility was used seven days for this user project.

Project 2038: R&D of High Pressure Hydrogen Storage System with increased Fire Resistance Rating

Background: High pressure H2-storage systems have to pass a so-called "Bonfire Test" during their licensing procedure in which their behavior in an external fire is investigated. The test is passed successfully if the tank (filled up to regular working pressure) completely releases the pressure without tank bursting. To achieve this so-called Thermally Activated Pressure Relief Devices (TPRDs) are utilized that open as soon as they detect a given temperature value (usually in the range of 120°C). Since the fire resistance of current tanks is not very high the diameter of the release opening of the TPRD is chosen rather large (4-5 mm) to provide for a quick depressurization of the tank. But this sudden hydrogen release might produce a so-called pressure peaking phenomenon that is strong enough to destroy surrounding enclosures like a garage, or, if ignited produces a H2 jet flame with a length of about 10-15 m that prevents any self-evacuation or rescue operations at a potential accident scene. To reduce the hazards arising from the tank depressurization the diameter of the TPRD should be reduced, but this leads to lower release rates and also to significantly longer depressurization times. These longer depressurization times in turn require a significant increase of the fire resistance of the tank which can be reached by different means.
To demonstrate experimentally the potential for a substantial increase of the fire resistance rating of Type IV hydrogen tanks using innovative thermal protection solutions and to collect data for the validation of numerical models and simulations the Warwick-Hexagon-Ulster User group applied for Transnational Access at KIT-HyKA to perform Bonfire Tests with thermally protected hydrogen tanks.

Results: The experimental work has not yet started since the tanks did not become available during the second reporting period. But during planning and preparation of the tests fruitful discussions and theoretical considerations on the test conditions unfolded between the user group and the host of the installation (KIT) as well as Pro-Science, who have extensive experience in performing such tests.
The experiments will be performed in the test vessel HyKA-A2. Since this vessel is designed for a static overpressure of 10 bar, Bonfire tests have to be performed under inert nitrogen atmosphere to avoid loads caused by the ignition of the hydrogen inventory of the tank as additional combustion load to the safety vessel. Due to the inertization a special burner system was developed at KIT that uses pre-mixed stoichiometric methane-air mixture as fuel. The fuel burns above a porous sintered metal plate that covers the upper face of the burner and prevents flame flash-back into the burner system. The complete burner system, from the flow meters for methane and air to the pore-size of the sintered metal plates is designed to fulfill the temperature profiles specified in the proposed "Global Technical Regulations" ECE/TRANS/WP.29/2013/41 that schedules maximum temperatures above 800°C (but below 1100°C) below the tank in a two-step test procedure. The burner system is capable of producing a maximum heat release rate (HRR) of approx. 165 kW, but considerations on experiments with car fires reported in literature led to the concern among the user group members that this HRR might be too low to be representative for a potential car accident scenario where HRRs in the range of 340 kW could be expected. Simulation calculations performed by the user-group member UU revealed that there is a significant influence of the HRR on the fire resistance of a tank, but simultaneously the HRR also affects the maximum temperatures reached below the tank considerably. The considerations and discussions are not yet completed and the results are planned to become an important contribution to WP9 (JRA3.1), where a "Harmonized methodology for testing of pressure relief devices of onboard pressurized hydrogen storage" will be formulated.

Project 2051: Behavior of hydrogen jet releases close to surfaces

Background: A free jet of a fluid entrains and mixes with its surrounding atmosphere as it flows

away from the nozzle. But with a surface on one side the entrainment is restricted and due to velocity differences a pressure difference across the jet occurs, deflecting it closer to the surface and, in the case of a flammable gas, increasing its flammable length. The behavior of surface jets is important for H2-safety since increasing flammable volume also affects the possibility of an ignition event. Also consequences for hazard analyses are possible since assumptions on the location of sensors, the definition of exclusion zones, etc. are based on the classical scaling laws and may not be valid under these conditions.
The properties of attached jets have been studied numerically, and to a lesser extent experimentally for unignited and ignited releases. In simulation calculations an interesting behavior was observed for the normalized overextent of the flammable distance from the nozzle induced by the presence of the surface. But because of the simple modeling assumptions that were used to enable the study these results require experimental confirmation.

Results: Experiments on quasi-stationary horizontal jets under variation of the distance to a surface whose normal is perpendicular to the jet axis will be performed KIT-HyKA. In the first part of the work optical methods (PIV, BOS) and concentration measurements will be utilized to determine the concentration distribution of He-jets along the jet centerline and the wall (either side or ground) as a function of the distance between the surface and the orifice. After scaling selected He-jet properties to H2 experiments with ignited H2-jets will be performed in the second part in a facility where combustion experiments are possible. In these experiments the influence of the distance to the wall and different ignition positions on the shape of the jet flame and the overpressures generated will be studied. As a reference additional experiments without wall will be conducted.

The main experimental series has not yet started since the Laser needed for the PIV-measurements of the first part of the work is under repair. During the discussion of the test parameters special attention was directed to the nozzle, since its shape has a significant influence on the flow characteristics of the effusing gas. To facilitate a comparison of the current work with experimental results and simulation calculations performed earlier it was agreed to copy the elaborated nozzle used before by members of the user group. The nozzle is fabricated and already installed to the test rig for the He-jets of the first part of the work.

Project 2023: Electrochemical properties evaluation of multi-layered thermal spray coatings for Solid Oxide Fuel Cells

Background: Of all types of fuel cells, Solid Oxide Fuel Cells (SOFCs) deliver the highest electrical efficiency. However, high production costs and poor durability are key barriers to the widespread commercialization of SOFCs. An approach to reduce costs and improve durability, which was introduced in this research, was to use a thin and cost-effective metal supported cell structure. The inclusion of metallic supports brings the advantage of high electrical and thermal conductivity, superior toughness, better thermal shock resistance, and good workability which are highly attractive attributes for SOFCs. The most common technique for fabricating anode-supported SOFCs is based on wet ceramic powder slurries, which are solidified by sintering at high temperatures. However, it is difficult to apply these wet ceramic fabrication methods to metal-supported SOFCs because the electrolyte sintering process could rapidly oxidize or densify the porous metallic supports. Therefore metal supported SOFCs based on thermal sprayed coatings are suggested. Plasma spraying high flexibility permits deposition of coatings with various microstructures and different thicknesses. Moreover the latest developments in plasma spraying (suspension and plasma spray thin film) bring unprecedented improvements in terms of possibilities to produce coatings with low thickness (from few to tenths of microns) and/or coatings with very low porosity (gastight), characteristics which were not available only few years ago and which are of key importance for high performance SOFCs. Suspension plasma spraying (SPS) is a modification of the traditional plasma spray process in which the feedstock powders are suspended in liquid for feeding to the plasma rather than being fed as dry powders with the use of carrier gas. The suspension permits the use of smaller feedstock powders (in the range of nano- and sub-micrometric size), why SPS can potentially produce much thinner and denser coating layers and more refined microstructural features than standard plasma spraying. It is also possible to mix in the liquid suspension two or more powder materials (e.g. Ni and YSZ) and then to produce homogenous and finely inter-dispersed anode layers with high density of triple boundary phases, which is a key issue in a high performance anode layer. A-SPS is the next generation of SPS method that brings improvements such higher process flexibility, higher deposition efficiency and improved process stability. Plasma spray thin film (PS-TF) is another technology that was recently developed. It is a low pressure plasma spray technology to deposit coatings out of the vapor phase. The vaporized coating material is transported in a hot and supersonic gas stream (2000-4000 m/s, 6000-10000 K) which is expanding in a 1 mbar chamber controlled atmosphere. This leads to high growth rates and the possibility to create homogeneous and gas tight coatings with thicknesses of 2 to 50 µm. The above mentioned technologies have been evaluated in our previous work and they showed very promising results, as they enable nano-sized feedstock material. However the evaluation of the functional layers, nano-structured coatings and fabricated single cells is at an early stage. A detailed electrochemical analysis is required to evaluate bottlenecks and to adapt the above mentioned technologies to the specific needs of metal supported SOFCs..

Results:

In 2014 the first of three planned testing campaigns for plasma-sprayed metal supported SOFCs has been performed. In this campaign 6 cells differing with respect to the anode and the electrolyte have been tested. Details about these cells are summarized in the following table.

test	**spray number**	**anode ID**	**anode state**	**electrolyte**	**cell ID**	**remarks**
1	14T0036	9	as-sprayed	70 µm TZP	7.182	
2	14T0032	1	as-sprayed	45 µm 8YSZ	7.183	
3	14T0034	5	as-sprayed	80 µm 8YSZ	7.184	durability test 250h
4	14T0032	2	oxidised	50 µm 8YSZ	7.185	
5	14T0036	10	oxidised	80 µm TZP	7.187	
6	14T0034	6	oxidised	85 µm 8YSZ	7.186	

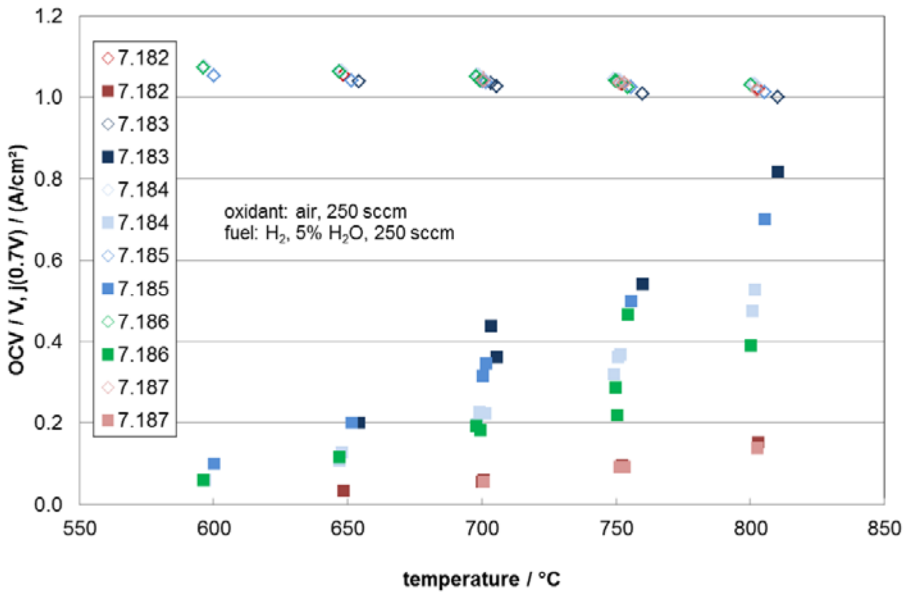

Figure 1: OCV and current density at 0.7 V cell voltage.

A summary of the results is given in **Figure 1**. All of the tested cells could be operated without any serious problems. None of the cells failed. The achieved OCV-values are ranging in between 1000 and 1040 mV at 750 °C whereas the theoretical value for the given gas composition would be 1087 mV. The difference has to be attributed to an imperfect gas tightness of the thermal sprayed electrolyte layer as well as a bending of the metal substrate. In comparison with other plasma sprayed cells tested in previous projects the observed gas tightness is acceptable. Nevertheless a comparison with state of the art anode supported cells, which usually exhibit a one order of magnitude lower leakage, shows that a further improvement of the electrolyte layer would be advantageous. The electrical performance of the analyzed cells strongly depends on the applied layers and materials. As no interdiffusion barrier layer was introduced in between the LSCF-cathode and the zirconia electrolyte, the performance is limited by the formation of an insulating strontium zirconate layer at the cathode electrolyte interface. Without an interdiffusion barrier layer a severe degradation of the cells is inevitable.

The analysis of the cells reveled a number of bottlenecks, which have to be eliminated in the next steps (ongoing cell development at University West):

1. selection of fully stabilized 8YSZ as the electrolyte (as both TZP samples showed the lowest performance)
2. application of a ceria based (Gd_2O_3 doped CeO_2) interdiffusion barrier layer onto the zirconia electrolyte to prevent the formation of strontium zirconate
3. further improvement of the electrolyte with respect to gas tightness
4. reduction of the electrolyte thickness (as the samples with the thinner electrolytes showed a higher performance)

2. Description of installations provided

- **HYKA:** At the hydrogen test centre HYKA of the Karlsruhe Institute of Technology (KIT) there are several safety vessels and test tubes for hydrogen combustion experiments in various scales and a large test chamber for hydrogen distribution and in particular combustion experiments. These vessels are: Hydrogen Test Chamber (PZ) and Safety-Vessels A1, A2, A3, A8.
- **MHtest:** The laboratory facilities for solid storage materials include state-of-the art and sophisticated instrumentation that allows accurate H2 storage measurements at different pressure/temperature conditions with a unique combination of chemical, volumetric, gravimetric and calorimetric analysis techniques. Chemical composition can be analyzed by an Elemental

Analyzer (CE Instruments). For H storage properties 3 specially designed PCT volumetric systems are available (0-150 bar, 300-700 K), a NETZSCH STA 409C Thermal Analyzer for combined TGA-.DTA-MS experiments, a SETARAM Thermal Analyzer for combined TGA-DSC-MS measurements and DSC experiments under reactive atmosphere up to 300 bar, a NETZSCH Phoenix high pressure DSC for high pressure calorimetry. Structural characterization is possible with a BRUKER Advance D8 X ray diffractometer, Moessbauer spectrometry (Fe, Sn), a Perkin-Elmer Spectrum GX FTIR spectrometer, a Perkin-Elmer Laser Raman spectrometer, several SEM microscopes (LEO Gemini) with EDX option and an ultra-high resolution

- **SOFCTEST (KIT-IWE):** At KIT-IWE a number of SOFC testing facilities are available for various material, electrode and single cell tests. The facility offered enables a detailed electrochemical characterization of single cells and repeat units under realistic operating conditions including:
  - Performance and stability tests
  - Electrochemical impedance spectroscopy and the impedance data analysis by the distribution of relaxation times
  - In-situ gas analysis (catalytic and electrochemical gas conversion along the gas channel) and the evaluation of reaction kinetic parameters
  - Analysis of the impact of fuel impurities as higher hydrocarbons, H2S and other gaseous species
  - Accelerated lifetime and cycling tests (load, thermal, redox, carbon deposition / metal dusting)

  The facility enables a complete characterization of a solid oxide fuel cell. The results can provide information about the individual loss mechanisms in the cell and their dependencies on operating conditions (temperature, gas composition, gas utilization and current density). Based on the results electrochemical models for the cell can be developed. Details concerning the testing methodologies and the related methods and models for data evaluation can be found in the references.

3. Services offered

At the hydrogen test site HYKA it is possible to investigate the whole spectrum of hydrogen phenomena: Research on different hydrogen sources and their distribution behaviour can be conducted, as well as experiments with different ignition sources. However, the most attractive feature of HYKA is the capability for well controlled, medium to large scale combustion experiments, covering all three combustion regimes (slow and fast deflagration and detonation). Therefore HYKA has been and is currently used in several EU projects (EIHP2, HYCOM, HySafe, StorHy, HYPER, …) and industry co-financed analyses (ICEFUEL, Vestolit,..).Two customers from German leading automotive industry conducted basic safety studies in the facilities for mitigation concepts and

Mainly for nuclear applications the structural integrity of pipes under the loads of radiolysis gas (stoichiometric hydrogen / oxygen mixture) detonations at initial pressures of up to 70 bar has been studies. For ITER fusion reactor safety studies and for the EC project StorHy the reactivity of different nano-scaled solid materials (metallic dusts) loaded with hydrogen have been basically studied with the unique dust explosion facilities of HYKA.

Apart from the experimental work KIT offers theoretical investigations and CFD-simulation calculations of the hydrogen phenomena mentioned above. The special tools GASFLOW, COM3D, DET3D and GP provide predictive capabilities as they have been intensively calibrated on the basis of broad experimental programs. So calculation for the experimental layout as well as for interpolations and extrapolations of experimental results are offered to the customer for the most efficient use of all resources.

Transnational Access @ CEA

Description of the infrastructure	
Name of the infrastructure: **CEA H_2 AND FUEL CELL ENERGY CHAIN INFRASTRUCTURE**	
Location (town, country): **CEA Centre de Saclay** 91191 Gif sur Yvette Cedex France	Location (town, country): **CEA Centre de Grenoble** 17 rue des martyrs 38 054 Grenoble Cedex 9 France
Web site address: www.cea.fr	
Legal name of organisation operating the infrastructure: CEA	
Location of organisation (town, country): Headquarters : Commissariat à l'Energie Atomique et aux Energies Alternatives Bâtiment Le Ponant D 25 rue Leblanc 75015 Paris (France)	
Annual operating costs (excl. investment costs) of the infrastructure (€): 2 911 410 €	

1. Running Transnational Access Activities

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2048: Performance evaluation of the XEN-5310 sensor and PCB connexion in an academic free jet/plume of helium in air

Infrastructure: GAMELAN experiment in FLOREAL

Description: One access granted to Xensor Inc to evaluate sensors improvements for He jet flow concentration measurements. Start of pre-test of PIV and concentration measurement during November 2014. Experimental campaign starting end of December to mid-january (first half of the campaign of 30 days of access). The second half will take place in Feb 2015 (when new sensors rulers will be received).

Analysis of the results and publication will follow (submission to ICHS 6).

Schedule: Program started as scheduled with a set-up of the experimental system. Old Xensor sensors placed for comparisons with the new ones. 2 part of the planning is also respected and took place in November. Set-up of the PIV laser system was done after having received the revised Yag laser system. Minimum flow rate enabling measurements was established at 10 Nl/min below which particle seeding is not enough anymore. The experimental matrix will be achieved in 10 days.

Different flow rates will be tested in a free jet configuration.

Slight modifications: This experimental campaign will be split in two campaigns of 5 days. The first one will test the sand alone sensors that CEA will receive 2nd week of December. The second half of test will be postpone to mid-February when ruler-sensors will be received by CEA. We are talking about modifications rather than delays since we already anticipated that the experiments had to be carried out in 2 or 3 parts. We knew from the beginning that the campaign could start within 6 months after acceptation of the user access. Nevertheless the process could not be carried out in one row for practical reasons (sensor design and production, FLOREAL Access planning etc....).

Amount of unit used: 40ke for 30 days of access. Access costs have been already described in appropriate documents.

Visits: CEA visited Xensor to discuss the feasibility of the access demand, Delft 204. Mission cost are detailed in official cost reports submitted by CEA DEN. Other communications with Xensor are done with mails and phone. As already mentioned in the access conditions to FLOREAL, CEA is performing the experiments and does not accept participation of the user. The user has specified his needs and CEA performed / is performing the experiments.

Dissemination: Publication to ICHS6 followed by submission to Int. Journal of H2 Energy.

2. Description of installations provided

- **Safety and risk assessment infrastructure - FLOREAL:** This installation offers 3 experimental set-up of 1 m^3, 41 m^3 and over 100 m^3 to measure release and dispersion of light gas (He, water vapour ...) in air. One of the strong interest of those facilities is that they are located in a relatively well temperature regulated warehouse (usually 1°C of variation over one day) and the set-up are even better regulated (the garage is close to a 0.1°C regulation). A second quality of the installation is that it is well instrumented (almost a 100 cathetometers, Pt100 sensors, PIV Yag laser, continuous laser and pulsed laser systems). Many previous experimental campaigns produced analytical results published in international journals.
- **Performance and Durability Infrastructure – In situ water repartition within operating polymer electrolyte fuel cells (EDIP/ Grenoble):** The EDIP installation and associated procedures allow determining the membrane water content in an operating fuel cell by small-angle neutron scattering1-8 using specific cells, transparent to neutrons (25cm2, golden aluminium plates with visualization holes along the gas channels and temperature controlled), and a mobile test bench (25 cm2 single cell capability, humidity and gas flow mass controlled and monitored, temperature monitoring from room temperature to 100°C). Operation conditions such as temperature or current densities can be varied over a representative range. data analysis protocol has also been developed to extract the water concentration profiles within the membrane during operation. These data can be used either to validate mass transfer models, to measure water transport at given humidity values or to study the water management in specific conditions. In addition all in situ experimentations are prepared using regular normalized test benches available at CEA/Grenoble.
- **Performance and Durability Infrastructure – Conductivity and MEasures of Dilatation of Hydrides (COMEDHY):** The equipment proposed, which is in operation since January 2010, enables hydrides materials to be tested in gaseous hydrogen pressures and temperatures. It is designed to understand how hydride behaves in terms of swelling and shrinking under mechanical constraints during absorbing and desorbing hydrogen. The vessel operates at pressures from 0 to 200 bar and temperatures up to 200°C, with an internal volume of 50ml-150ml of hydride. The influence of the applied mechanical force can be assessed during a few tens of absorption/desorption cycles. A full temperature measurement inside the hydride and the vessel allows establishing the link between mechanical stresses between the hydride and its container and the thermal efficiency of the hydriding reaction. The current method for measuring the absorbed volume is based on the use of mass flow devices. As a consequence, the experiment needs a minimum mass of hydride, absorbing at least 10 Nl of hydrogen. Improvements proposed in JRA2 will allow coupling a volumetric (Sievert) measurement method to the

experimental vessel and modifying the test bench in order to increase the resolution of the installation and to accurately deal with lower absorbed volumes.

3. Services offered

- **Safety and risk assessment infrastructure - FLOREAL:** This installation provides a unique scientific environment with strong international links and commitment of the research team assessed by:
  - An active participation to European projects and French National projects in the area of Hydrogen Safety. For example during the recent years, the team has been active in HYSAFE, NATURALHY, HYPER, HYAPPROVAL co-funded by the EC and DRIVE, HYDROMEL co-funded by the French National Research Agency. In addition, the nuclear hydrogen safety supports long term research in the frame of SARNET 6th and 7th PCRD NoE or SETHII OECD.
  - A contribution to the management and the works of the HySafe International association and IEA-HIA annex 19 group of expert.
  - A constant attraction of young researchers from different countries

  The quality of results obtained in this installation relies on the application of "Good Laboratory Practice" that have been certified ISO 9001 v2000. On this basis clear operational procedures are made available, particular attention is given, to reproducibility and to the data analysis and storage for further use. In addition, a strong modelling team is committed to support the design and analysis of experiments. This team of 10 permanent researchers in modelling and simulation of hydrogen distribution and combustion work in strong interaction with the experimental team. The models are implemented in the CEA in-house code CAST3M or CHOMBOCLAW developed in collaboration with Lawrence Berkeley Laboratory (CA,USA). Both codes are open source codes and can be used by external teams. During the course of HySafe, these codes have been used and partially qualified for hydrogen. They will provide a strong contribution to the effort toward a European modelling tool presented in WP10 (JRA4).

- **Performance and Durability Infrastructure – In situ water repartition within operating polymer electrolyte fuel cells (EDIP/ Grenoble):** The EDIP team is composed of experienced engineers and technicians who will help in the redaction of proposals for neutron beam-time allocation by any large facilities (for example ILL (Grenoble) or LLB (Saclay) in France). Upon acceptation the EDIP team can help for integrating polymer into a fuel cell, especially with adapted electrodes, and validate the performance of such a new Membrane Electrode Assembly (MEA). This can be done in a specific laboratory at CEA/Grenoble allowing fuel cell assembling which comprises tape casting and spray facilities to fabricate active layers, a press and ovens to assemble the polymer with two adapted electrodes. The EDIP team can also offer preparing the experimentation and positioning innovative materials regarding current state of the art in normalized conditions, by using normalized test benches. Owing to the safety rules of the installation and to its complexity (H_2, mobile test bench) and its environment (under neutron flux), the in situ experimentation will be conducted by the EDIP team assisting the research team. Support will also be provided for data analysis, and in particular the use of the unique neutron small angle scattering data analysis protocol that allows extracting water profile concentration. After adaptation of the technique to X-rays, the EDIP team will also allow the access to a SAXS camera either to run the experiments directly in the laboratory or to prepare experiments that can be run in parallel on a synchrotron (again after submission of a proposal for beam-time). Open access will be provided to the EDIP installation for maximum 20 days per year with minimal time periods of a week. The time allocation in the year will depends on the answers of neutrons and X-RAYS large facilities. It is worth noting that each in situ experimentation time (from 3 to 5 days) will be systematically associated with 9 to 15 days time preparation and data analysis in the fuel cell laboratory at CEA/Grenoble. This results in a full experimentation in the EDIP installation ranging between 12 to 20 days 25% of the time being spent under the neutron beam.
- **Performance and Durability Infrastructure – Conductivity and MEasures of Dilatation of**

Hydrides (COMEDHY): Open access will be provided to the COMEDHY installation for maximum 40 days per year with minimal time periods of two weeks. The research team will be offered assistance by the COMEDHY team during all its stay. The COMEDHY team is composed of 3 experienced engineers respectively in mechanical testing, mechanical modelling and thermal modelling, 1 technician for the installation. The COMEDHY team will help for proposal preparation. Upon acceptation under the rules of WP1, and owing to the safety rules of the installation, a team composed of experienced engineers and technicians will assist the research team in the whole process of preparing and performing hydride swelling tests under hydrogen. The COMEDHY team will also help in analysing all the obtained results.

Transnational Access @ IFE

Description of the infrastructure
Name of the infrastructure: JEEP II
Location (town, country): Kjeller, Norway
Web site address: www.ife.no
Legal name of organisation operating the infrastructure: Institute for Energy Technology (IFE)
Location of organisation (town, country): Kjeller, Norway
Annual operating costs (excl. investment costs) of the infrastructure (€): no figure given

1. **Running Transnational Access Activities**

Five project has been executed in the 2nd period; all on installation 1, PUS:

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2009: Li-disorder in the hydrogen storage material LiCe(BH4)3Cl and detailed investigation of hydrogen positions

There has been conflicting reports on the amount of Li disorder in the phase $LiCe(BH_4)_3Cl$. High Li disorder is interesting from the perspective of favourable kinetics for hydrogen sorption reactions relevant for hydrogen storage and for the possibilities of using the material as a solid state Li ion electrolyte. Powder neutron diffraction (PND) measurements were performed both at ambient temperature and at 8 K during *22 days* from March 13 to May 7. The experiments were carried out by IFE staff and did not involve any traveling. The measurements indicate the Li ions are disordered and that the disorder persists to very low temperatures (8K).

Project 2026: Hydrogen positions in magnesium borohydride ammoniates

Metal borohydride ammoniates $(M(BH_4)_n xmNH_3$ contain both protonic and anionic hydrogen. This could lead to new concept to tailor the stability of hydrogen storage materials, however, a proper understanding about the dihydrogen interactions ($H^{\delta+}$ --- $H^{\delta-}$) is needed. Several metal borohydride ammoniates have been structurally characterized by X-ray diffraction lately, without obtaining reliable information about the hydrogen positions due to the small scattering power of light elements. In this project, two magnesium borohydride ammoniates, $Mg(^{11}BD_4)_2x6ND_3$ and $Mg(^{11}BD_4)_2x2ND_3$ have been prepared with isotopes suitable for neutron scattering. The materials were measured at the PUS installation for 20 days in the period from May 15 to May 26 without user visit. Structure analysis is in progress.

Project 2029: Synthesis of new hybrid hydrides by combination of anionic borohydride and imidazolate ligands: the first member, Li(BH4)Im

Porous metal hydrides could be of interest for hydrogen storage due to their possibility to adsorb significant amounts of hydrogen in addition to the hydrogen contained in the crystal structure. The user has synthesised the first porous hydride based on the bridging ligand imidazolate (Im,

[$C_3N_2H_3$]-): Li_2ImBH_4. X-ray diffraction does not yield reliable information about orientations of the BH_4 tetrahedra, which is essential to understand the coordination behaviour of BH4 which will aid the synthesis of similar compounds. A with for neutron diffraction with ^{11}B and deuterium was synthesised and measured at the PUS instrument for 13 days in the period May 9 to May 21 2014. The user did not visit the facility. Preliminary results were presented at the 14th International Symposium on Metal-Hydrogen Systems, MH2014, in Manchester, UK.

Project 2040: Structural characterization of Sr(11BD4)2.2ND3 by powder neutron diffraction

The project is closely related to project 2026 and concerns crystal structure determination of the metal borohydride ammoniate $Sr(^{11}BD_4)_2.2ND_3$. Powder neutron diffraction data were collected at the PUS instrument for 7 days in the period May 26 – June 1 without user visit. Data analysis is in progress.

Project 2062: The Crystal Structure of NdGaDx

The alloys NdGa can absorb hydrogen. In addition to function as a hydrogen store, the material shows a magnetocaloric effect which may be tuned by the hydrogen content. Knowledge of the hydrogen site preference, which is only accessible with neutron diffraction, is crucial for to understand and tune the structure-property relations.
A deuteraded sample was prepared and measured along with the deuterium-free alloy for 14 days at the PUS instrument in the period September 19 to October 10 without user visit. Data analysis is in progress.
Several additional projects are planned or in progress. Project 2066 is on-going. This project requires the possibility to expose the material hydrogen and/or deuterium gas at low temperatures, which was not possible at the time when the application was received. However, the cryostat has been modified to allow gas introduction to newly designed sample holder, thus greatly increase the usability of the facility to investigate hydrogen adsorption on materials with high specific surface area. Projects 2030 and 2032 have been approved but the users have not yet managed to prepare samples in sufficient quantities. For projects 2031, 2041 and 2061, samples are received from the users but the measurements have not yet been performed to capacity limitations. Project 2027 is difficult to perform according to the user's needs and will most likely not be completed.

2. Description of installations provided

- **Installation 1 – Powder neutron diffractometer, PUS:** The high-resolution powder neutron diffractometer PUS has been in operation since 1997 and has been extensively used for crystal structure determination of hydrogen storage materials. The sample can be cooled or heated in the temperature range 8-1300 K and exposed to reactive gases (e.g. hydrogen gas) at ambient and elevated temperatures.
- **Installation 2 – Powder neutron diffractometer, ODIN:** The flexible powder neutron diffractometer ODIN is in the final stage of construction and will be operational from 2011. Due to a larger beam channel and the much larger detector area compared to PUS, the data acquisition times will be drastically reduced at improved resolution which will allow for in-situ type measurements.
- **Installation 3 – Small angle neutron scattering, SANS:** The SANS instrument was built in 1986 and has been upgraded several times since then. It uses neutrons from a cold moderator

3. Services offered

The beam time at the neutron scattering instrumentation at the JEEP II reactor is 100% managed by the researchers at IFE. Access to the instruments is currently available to external users through collaboration with the IFE researchers or through a formal application. In the last couple of years (2008-2009), approximately 20 external research groups from Norway, Europe, US and Japan have been involved in neutron scattering measurements at JEEP II. Facilities are available for sample handling under inert conditions. The JEEP II reactor is typically in operation 250 days/year with 3 scheduled yearly stops for maintenance.

4. Description of work

- **Access activity 1 – Powder neutron diffraction:** Activity plan:
  a) The sample is brought to IFE by the user. The technical staff will assist the user in mounting the sample in the appropriate sample cell, under intern conditions when necessary. In rare occasions, the sample can be sent to IFE for measurement. This requires thorough prior communication on e-mail or telephone between the user and IFE staff to ensure that the measurement if performed according to the user's needs.

  b) The user may stay at IFE for a period of time after the measurement, typically 2-5 days, for help with the data analysis. IFE has staff with expertise in powder crystallography which can aid in all the steps from indexing to structure solution and refinement. If the data analysis is not finished by the end of the stay, the user will be advised on how to proceed further at home. The user will be followed up and further guided by email or telephone when appropriate, until the data analysis is completed. The IFE staff will help writing the relevant parts of the manuscript when the results are published.

- **Access activity 2 – Small Angle Neutron Scattering:** The activity plan is similar to that described in "Access activity 1 – Powder diffraction". The user will be guided by experienced personnel through preparation and execution of the measurement as well as data analysis and dissemination.

Transnational Access @ HSE

Name of the infrastructure: **Health and Safety Laboratory**
Location (town, country): **Harpur Hill, Buxton, UK**
Web site address: **www.hsl.gov.uk**
Legal name of organisation operating the infrastructure: **Health and Safety Executive**
Location of organisation (town, country): **Buxton, UK**
Annual operating costs (excl. investment costs) of the infrastructure (€): no figure given

1. Running Transnational Access Activities

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2046: Characterization of high pressure hydrogen release flammability profiles

The characterisation of high-pressure hydrogen releases has been completed and reported to H2FC and the user group in report 2046. The work undertaken was completed using the High Pressure Hydrogen Facility at HSL. The project was planned in collaboration with the consortium lead, Professor P Bernard (Université du Québec) in a series of teleconferences. The test plan is detailed below. Four separate test series were proposed:

- Unignited experiments of high pressure H_2 jet releases close to the ground (SERIES 1)
- Ignited experiments of high pressure H_2 jet releases close to the ground (SERIES 2)
- Unignited experiments of high pressure H_2 jet releases close to a ceiling (SERIES 3)
- Ignited experiments of high pressure H_2 jet releases close to a ceiling (SERIES 4)

For each series performed, six configurations were investigated with two repeats of each configuration. Two different flow conditions were chosen to give similar distances to the lower flammable limit (LFL) but through differing nozzle sizes and pressures. A flow rate of 6-8gs^{-1} was anticipated to give an estimated distance to the (LFL) of 4-5m for a free jet. The hydrogen reservoirs were known to decrease in pressure during each test, which was between 20-40 seconds long, the final pressure being approximately 90% of the initial pressure. Table 1 , 2, 3 and 4 describe the completed tests.

Table 1: TEST 1 - Unignited releases of high pressure H_2 close to the ground

Test Pressure (barg), Orifice Size (mm)	Distance from ground (m)								
	0.05			0.48			1.22		
150, 1.06	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
425, 0.64	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)

Table 2: TEST 2 - Ignited releases of high pressure H_2 close to the ground

Test Pressure (barg), Orifice Size (mm)	Distance from ground (m)								
	0.05			0.48			1.22		
150, 1.06	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
425, 0.64	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)

Table 3: TEST 3 - Unignited releases of high pressure H_2 close to a ceiling

Test Pressure (barg) , Orifice Size (mm)	Distance from ceiling (m)					
	0.08			0.49		
150, 1.06	(1)	(2)	(3)	(4)	(5)	(6)
425, 0.64	(10)	(11)	(12)	(13)	(14)	(15)

Table 4: TEST 4 - Ignited releases of high pressure H_2 close to a ceiling

Test Pressure (barg) , Orifice Size (mm)	Distance from ceiling (m)					
	0.08			0.49		
150, 1.06	(1)	(2)	(3)	(4)	(5)	(6)
425, 0.64	(10)	(11)	(12)	(13)	(14)	(15)

The schedule for the tests was a 4 week window as stipulated by the H2FC Trans National Access rules. This four week window was used to perform all the test specified in the plan and provide a report to the consortium. The test schedule ran to time but did encounter some difficulties with the hydrogen measurement as one sensor did not function correctly; this reduced the expected number of measurement locations down to four from five. Electrical issues also affected the recording of some of radiometer measurements and some data was corrupted. The user group consortium did not visit HSL. Number of Units used: 4.

2. Description of installations provided

The High Pressure Hydrogen Facility (HiPress) is capable of compressing, storing and releasing hydrogen at pressures up to 1000 bar. The system is engineered for high flow rates of hydrogen and can be fitted with exit orifice sizes to a maximum of 9.5 mm. The facility consists of a manifolded hydrogen delivery system and purge gas cylinder packs. The hydrogen is fed into a gas booster, which increases the pressure of the gas up to 1000 bar and can be stored in two 50 litre capacity storage vessels.

The High Pressure Hydrogen Facility was fully untilised for the project 2046 as a high release rate and starting pressure of hydrogen was required with two small orifice exits. HSL were able to modify the release system to allow surface jets to be studied. The modification to the release also included the creation of an elevated ceiling. This may be useful to other consortiums as HSL can now study the effect of a ceiling on high pressure hydrogen jets (ignited or unignited) as was undertaken for project 2046.

3. Services offered

The infrastructure at HSL offers a wide range of possibilities for studying different phenomena associated with hydrogen safety. HSL has facilities enabling the investigation of many of the practical safety questions associated with the hydrogen economy, such as; what happens if there is a leak at a hydrogen fuelling station or on a hydrogen vehicle, how does spontaneous ignition occur and how can it be controlled and what happens to hydrogen-handling equipment under impact. This coupled with HSL's wide range of measurement and sensing techniques, which can be incorporated as part of the planned work on these facilities, enables us to address the whole spectrum of hydrogen safety related research.

HSL has carried out a large amount of past research into hydrogen safety giving us a good working background and an appreciation of the safety and practical concerns with carrying out hazardous testing. Clients for this work have included the EU, in projects including HySafe, HYPER and NaturalHy, the UK government and various industrial partners including automotive companies, and the nuclear and aerospace industries.

As well as the experimental facilities, HSL also has a mathematical sciences unit, which carries out CFD-simulations of the hydrogen phenomena mentioned above using a number of different software packages to predict hydrogen distribution and combustion behaviour.

Transnational Access @ JRC

Description of the infrastructure
Name of the infrastructure: JRC Hydrogen and Fuel Cell Test Facilities (JRCH2&FCTF)
Location (town, country): Petten, the Netherlands
Web site address: http://ie.jrc.ec.europa.eu/
Legal name of organisation operating the infrastructure: European Commission, Directorate-General Joint Research Centre, Institute for Energy
Location of organisation (town, country): Petten, the Netherlands
Annual operating costs (excl. investment costs) of the infrastructure (€): 1,500,088 Euro (as of 2007)

1. Running Transnational Access Activities

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2004: The performance testing of the hydrogen sensor based on polyaniline

Background: Within this project, the user intends to observe the operation of the hydrogen sensor under various conditions. For this purpose, the user proposes to make a number of tests of 3-5 resistive sensorsprepared under different conditions (e.g. the time of plasma treatment).

The sensors are prepared on silicon substrates (chips ~2 mm x 2 mm) and can be mounted on standard TO5 packages (with no caps, of course). The base-line resistance is ranging from 1 to 500 kOhm. Testing voltage and current may not exceed ~2 V and ~0.1 mA, respectively.

Results: During one week of access to the SenTeF infrastructure several tests on the gas sensors were carried out: calibration test (200–1000 ppm of hydrogen), humidity test (0–80 %) and temperature test (20–80 °C). Using the separated measurement workplace also response time test (to 1% of hydrogen) and cross-sensitivity test to gaseous ammonia were performed. Due to some measurement difficulties, data acquired from SenTeF did not correlate with measurements at user. Thus, further work is required to improve the sensor performance and measurement repeatability.

Since user's samples were only research prototypes, they did not provide a readout circuits with their sensors. Thus, the main difficulty was the electrical measurement. High baseline resistance and limited measurement voltage made data acquisition very difficult, since SenTeF facility was designed mainly for industrial standards.

Project 2005: Performance range of a catalytic oxyhydrogen sensor device

Background: At the present stage we would need the support of the H2FC testing facilities for the following purposes:

- Determination of the performance range of our H2 sensor under different environmental parameters, i.e. temperature, humidity and pressure in order to quantify variations in sensitivity, response time (t90, t50), linearity, hysteresis and reproducibility
- Check of the cross sensitivity to gas mixtures (N2, CO2) and the sensitivity changes under different Hydrogen /oxygen concentrations
- Test of the impact of chemical species which are known to have detrimental effects on sensor life like H2S, CO, H2SO4 (spray) and gaseous silicon compounds

With some of these results we would attain the basic knowledge to fulfil standard performance requirements for stationary H2 detection (ISO 26142) i.e. a step forward to achieve a certification.

Results: a brief summary of work carried out is described below:

- Short term stability, linearity and accuracy (three sensor prototypes exposed to 0 vol%, 1 vol% and 2 vol% H2 concentrations)
- Temperature dependence (the H2 sensitivity for three sensor prototypes was tested at 5 , 20 and 35°C)
- Response and recovery time at 1% H2, 20°C and 0% RH.
- Pressure Test: sensitivity at 100, 110 and 120 kPa (0.5 and 1% H2)
- Relative humidity (RH) effect: Sensitivity at 20, 50 and 80% RH (0.5 and 1% H2) simultaneously measured for three prototypes
- Cross sensitivity to poisonous gases CO, H2S and SO2

There were difficulties to achieve low temperatures (T< 0 °C) with the three prototype sensors in the test chamber (due to the sample masses to be cooled and the heat generation of the circuit boards). The measurement was repeated by the SenTef personnel with one sensor in the chamber the week later. There were sporadic difficulties with the PC data acquisition of the sensor CAN bus signals Through these testing results we have gained an extended knowledge on the performance of our sensor under different environmental conditions. A 5 day stay visit was needed.

Project 2054: Effect of surface treatment on the long-term cyclic stability of BCC hydrites in pure hydrogen

Vanadium-based hydrogen storage materials with a body-centered cubic crystal structure show a high gravimetric and volumetric hydrogen density at ambient pressure and temperature conditions. This makes them promising materials for stationary and mobile hydrogen storage applications. Two factors yet limit their practical use: 1) High raw material cost, and 2) low tolerance towards impurities, which may be contained in the gas phase, such as oxygen, moisture, CO2 etc. The low tolerance of metal hydrides towards impurities in the gas phase reduces the efficiency of a solid hydrogen storage material and may even prevent hydrogen sorption reactions. It is related to the reactions of gas phase impurities occurring at the surface of the metal hydride, e.g. the formation of surface metal oxides during cycling with hydrogen containing oxygen. These metal oxides block the active sites of the metal, which promote hydrogen adsorption and/or dissociation. Additionally, they may act as a diffusion barrier for the hydrogen atoms on their way to the bulk of the metal hydride.

At the Karlsruhe Institute of Technology (KIT) a novel method for the modification of the surface of BCC alloys is currently under development. The aim is to improve the tolerance of BCC hydrogen storage materials towards impurities in the gas phase. This will be achieved by selectively removing those metals from the surface of the material, which show a strong tendency to form metal oxides. Only those metals shall remain at the surface of the material which show a lower tendency to form a metal oxide. It is expected that the tolerance of the BCC hydrogen storage material towards impurities can hereby be considerably be improved.

An apparatus for the accurate determination of the hydrogen storage capacity and uptake kinetics is available at KIT and ready to be used for transnational access. However, it is not suitable for the determination of the long-term cyclic stability due to the lack of automated measurements. The Joint Research Center (JRC) has built up an apparatus, which can be used to determine the long-term stability of the metal hydride under the influence of impurities. We would like to determine the long-term cyclic stability of the unmodified BCC material and two modified materials with this apparatus. The impurity gases which shall be used are oxygen and water. Each measurement shall be performed with 1000 ppm oxygen and 200 ppm water for several hundred cycles.

Project 2059: Hydrogen storage measurements in grapheme-based materials

The present project aims at a collaboration with JRC Petten [SolTeF] to characterize such innovative nanostructures of Graphene in terms of physical and chemical properties, including the H2 storage thermodynamics (enthalpy, entropy of reaction or interaction, equilibrium pressure...) and kinetics aspects. Such investigations need highly accurate volumetric or gravimetric techniques in a wide range of pressure values (from vacuum to 150-200 bars) and temperature (77-300K or more) in order to cover as much as possible chemisorption and physisorption characteristics of our materials.

FBK has a wide expertise and facilities for surface characterization (XPS, UPS, Tof-SIMS, AFM), as well as hydrogen sorption measurement and testing set-up. In FBK a suitable and innovative instrument for in-depth studies of hydrogen sorption is being developed, based on a differential pressure apparatus (DPA), which allows volumetric measurements with an approach different from the standard one for this kind of measurements, by using a highly accurate differential pressure transducer.

FBK intends to collaborate with JRC laboratory SolTeF within the FET Flagship Graphene project to have expert support for material validation and access to the extended characterization facilities. The present application is specifically aiming at:

- Utilizing instruments and the available expertise in H2FC to characterize and validate the different graphene-based materials produced and studied in the frame of FET Flagship Graphene project, in particular: characterization of hydrogen physisorption, in the typical range of pressure (0-150/200bar) and temperature (77-300 K) for carbon based materials. Both gravimetric and volumetric measurements are useful for such materials.
- Comparing the results with the ones obtained with a homemade instrument and validate them, in terms of accuracy and sensitivity.

Project 2060: Environmental characterization and Safety Test of an Hybrid Power Source combining a LT-PEMFC and a Lithium Ion Battery Energy Storage embedded into a passive cooling shelter for off-grid Residential, Telecom and Agriculture applications

The aim of the project is conduct environmental, functional and safety test on an innovative hybrid power system designed and realized by Genport. Within this project a set of functional tests with different combination of electrical loads and RES configurations will be conducted in order to characterize the behaviour of the system in wide temperature range from 5 C to 40 C. Additionally safety tests will be executed in order to evaluate the fail-safe behaviour of the hybrid power source components as well as from the safety point of view, the interaction of

the different sub-system (Fuel Cell Stack, Balance of Plant, ion lithium cells) will be investigated. A final task will be the analysis of the framework of norms among the existing in order to identify the most suitable set for the next CE certification. The project will be structured into four main activities: Definition of the test specification, Setup of the test equipment's, execution of the experiments and analysis of the result.

2. Description of installations provided

- **Hydrogen fuel and air Quality test Facility (H2QF):** The Hydrogen fuel and air Quality test Facility (H2QF) which is just commissioned by JRC, is composed of dedicated high purity gas supply lines connected to a 3 kW PEFC/DMFC test station combined with a dual (EI/IMR) mass spectroscopy gas analyzer to quantify and monitor FC feeds and emissions for the assessment of the effects of hydrogen fuel quality and air contaminants on the performance (and degradation) of single cells and short stacks. This includes the establishment of cross contaminant effects which is a too least studied scientific issue to urgently identify tolerable fuel quality of hydrogen when produced by different production methods (electrolysis, reforming, gasification, fermentation, etc) and to refine hydrogen fuel specifications (for fuel cell use). Apart from resolving such scientific bottleneck, the installation enables simulation of different operation regimes such as temperature (> 160°C), pressure, gas composition, humidity etc in addition to current distribution measurements using segmented cells and electrochemical impedance measurements all accessible for users in this WP.

- **Environmental Test Chamber (ETC):** The Environmental Test Chamber (ETC) comprises a unique combination of a walk-in environmental chamber housing a six-degrees-of-freedom (6DoF) computerized vibration test system with provisions foreseen to test up to 100 kW fuel cells or other test items. The environmental conditions include the simultaneous application of multi-axial shocks & vibration at frequencies of up to 250 Hz (i.e. field test data), ambient temperatures in the range of -40°C to +60°C and 15-95% relative humidity of the ambient. Such environmental conditions are most relevant for hydrogen and fuel cells in transport application including the use of portable and mobile devices as well as the safe transport of stationary devices to and from the installation site. The design and the safe, reliable and durable operation of such devices in these applications require the definition and experimental validation of test protocols and the testing (e.g. structural integrity) under a variety of environmental conditions (ambient temperature & humidity, vibrations & shocks). This is a too much neglected research field (scientific bottleneck). Moreover, this chamber is equipped with safety features such as gas sensors and ex-proof surveillance cameras linked to a video recording device.
- **Hydrogen production Performance characterization Facility (H2PF):** The Hydrogen production Performance characterization Facility (H2PF) comprises of a small scale hydrogen production unit (reformer) based on reforming of low calorific natural gas to high purity hydrogen as fuel cell feed and an online monitoring of reformer input and output fluid and energy streams to survey quality of feeds and products and to determine performance in terms of efficiency and CO2 emissions under various operation modes. So far, very little research is ongoing to support the definition of test protocols and their experimental validation on the energy conversion and environmental performance of small scale reformers (scientific bottleneck).
- **Solid-state Hydrogen Storage Testing Facility (SolTeF):** The Solid-state Hydrogen Storage Testing Facility (SolTeF) lab is dedicated to measure the hydrogen sorption parameters employing commercially available, state-of-the-art experimental set-ups based on volumetric, gravimetric and spectrometric methods. What makes SolTeF rare is first of all its independency from material development centres, and its focussing on accuracy and repeatability of hydrogen sorption measurements and on the parameters influencing them. The laboratory is on its way to accreditation and offers the widest range of techniques available for this type of analysis. In addition, it offers long-term materials and systems stability studies, which could saturate any “standard” laboratory. The experimental conditions can vary in the range from 77 K to 723 K and pressure up to 200 bar. Equipment for samples handling and storage under inert atmosphere is available. SolTeF delivers “second opinion” analyses, independent of any third party interests. The long term objective of the laboratory activities is development of best practices and standards for hydrogen absorbing materials testing. The laboratory is also focused on reviewing existing measurement standards, identifying the key experimental parameters mostly affecting the quality of the results and benchmarking performance of various techniques for sorption measurements. Finally, the cross-world inter-laboratory comparisons have been organised by the SolTeF, the first ever of this type..
- **High-pressure hydrogen tank testing facility (GasTeF)**: The High-pressure hydrogen tank testing facility (GasTeF) can simulate hydrogen and methane fast filling cycles in any type of high pressure containers. GasTeF is a unique facility among the publicly funded research centres in Europe, because it can test under real conditions full scale high-pressure components using hydrogen as a medium. It consists basically of a two-stage compressor and a testing chamber equipped with temperature control and hydrogen tank diagnostics such as thermocouples, pressure gauges and a gas chromatograph. These components are installed in a safety bunker filled with nitrogen during experiments and protecting the external environment from every possible accidental situation. The facility is designed to be able to fill in an

hydrogen container to a pressure 880 MPa within 3 minutes and to slowly empty it. Permeation rates an temperature evolution can be continuously monitored and life cycle studies can be performed. By making use of the computational facility HyMode numerical simulations (Computational Fluid Dynamics) of fast filling and decompression modes are performed complementarily to the experimental campaign. The overarching objective of GasTeF is the support to the development of performance and safety standards for the high pressure hydrogen components.

- **Sensor Testing Facility (SenTeF):** The Sensor Testing Facility (SenTeF) is the only sensor testing facility in Europe dedicated to the independent performance assessment of commercial hydrogen sensors and sensors in development. Testing conditions can be controlled with high accuracy and sensor performance is evaluated impartially allowing different sensing technologies to be compared directly. The SenTeF facility simulates typical and atypical working conditions for hydrogen safety sensors. As a key enabling technology for safety monitoring of hydrogen applications hydrogen sensors are assessed using the SenTeF facility for accuracy, sensitivity, response and recovery time under various conditions of humidity (0 - 90% RH), temperature (233 - 393 K) and pressure (0.7 – 1.3 bar). Cross sensitivity to contaminant species and sensor long term stability are also assessed. We have been involved in impartial hydrogen sensor performance testing since 2001. In the execution of our activities we collaborate with sensor developers, manufacturers, end users, renowned research institutions and international standards organisations to support the safe use of hydrogen also through the development of standards for hydrogen sensors and guidelines for their correct and effective use.

3. Services offered

- **Hydrogen fuel and air Quality test Facility (H2QF):** This new facility can be used by the selected research fellows and visiting scientists. All installation features as described above are offered to these users. The technical and scientific support offered is described in the description of work. This installation will be used in a FCH JU funded project to study the effects of hydrogen fuel and air quality including cross contaminant effects on fuel cell performance to refine the tolerable upper limits of common contaminants. Eventually this research work will provide experimentally verified inputs to a revised ISO standard on hydrogen fuel specification applicable to fuel cells.
- **Environmental Test Chamber (ETC):** This facility can be used by the selected research fellows and visiting scientists. All installation features as described above are offered to these users. The technical and scientific support offered is described in the description of work. Most interesting scientific achievements obtained by users in concern to the offered installations: This facility was used in the FP7 CELINA STREP to conduct vibration tests on a 3 kW PEFC.
- **Hydrogen production Performance characterization Facility (H2PF):** This new facility can be used by the selected research fellows and visiting scientists. All installation features as described above are offered to these users. The technical and scientific support offered is described in the description of work. It will be used in a FCH JU funded project to study the effects of hydrogen fuel and air quality including cross contaminant effects on fuel cell performance to refine the tolerable upper limits of common contaminants. Eventually this research work may lead to a revised ISO hydrogen fuel standard..
- **Solid-state Hydrogen Storage Testing Facility (SolTeF):** SolTeF offers analytical services for assessment of hydrogen solid-state storage parameters such as overall capacity, pressure-composition isotherms (PCI), thermal gravimetric analysis coupled with mass spectrometry (TGA-MS), thermal desorption spectroscopy (TDS) and reaction kinetic curves. Long term cycling behaviour studies are also possible using an in-house developed device. The laboratory has been created upon suggestion of EC DG RTD, to guarantee independent assessment of experimental results, and to meet the needs of two types of users: laboratories developing advanced hydrogen storage materials but not possessing sorption characterisation techniques, and laboratories capable to measure hydrogen capacities, but requiring an independent “second opinion". Since 2007 the laboratory delivered independent assessments of hydrogen sorption

parameters to both types of customers in the frame of EC co-financed FP6 projects HYTRAIN and NESSHY. The laboratory has developed a database for archiving and comparison of row experimental data, which is the unique feature in the world scale. Access to the database is controlled by passwords in order to guarantee confidentiality and respect of Intellectual Property Rights (IPRs). Various European projects (COSY, HyStore) have already used SolTeF for hydrogen capacity and kinetics measurements in the years 2009-2010. Peer-review publications have been submitted but not yet published**.**

- **High-pressure hydrogen tank testing facility (GasTeF)**: GasTeF will offer the high pressure dynamic and static testing of hydrogen and methane high pressure containers. In this project, GasTeF will in particularly offer experiments for a detailed understanding of the physical-, thermal-, chemical and/or strain processes underlying the component deterioration mechanism, and therefore address not only car component manufacturers but also, and especially, R&D research centres. Due to the fact that commissioning has just finished, GasTeF activities have not yet produced scientific achievements and could not yet absolve customer requests, but there is a considerable interest to access the facility by various manufacturers of hydrogen and methane high pressure components, as well as by national regulatory bodies.
- **Sensor Testing Facility (SenTeF):** SenTeF offers accurate testing of commercial hydrogen sensors and sensors in development. Analyses of test results are used to provide a performance assessment which is independent of sensor manufacturer interests and which can be used to impartially compare sensing technologies for end users and other interested parties..

4. Description of work

Modality of access under this proposal:

The modality of access to the JRC installations (and service) offered in this WP will follow the procedures to be established under the activities of WP2 [N1]. The actual access will also be governed by the JRC site security clearance provisions and the applicable law and regulations including environmental and safety rules.

Support offered under this proposal:

As far as the various JRC installations (and service) of this WP are concerned, technical assistance by JRC staff is provided to the user throughout the test campaign in particular to prepare the tests and to operate the equipment; where appropriate training will be given. The test results are discussed with scientists of the Institute which have a background in a variety of fields including chemistry, physics and engineering. This includes assistance on the evaluation and the presentation of the results of the test campaign in peer-reviewed publications. The facilities are usually operated by minimum one technical staff and one scientific staff. Administrative and infrastructural assistance and support on health, safety and environment issues are provided by the JRC administration.

Transnational Access @ Jülich

Description of the infrastructure
Name of the infrastructure: SOFC Durability Experimental Facility (DurSOFC)
Location (town, country): Jülich, Germany
Web site address: www.fz-juelich.de
Legal name of organisation operating the infrastructure: Forschungszentrum Jülich GmbH
Location of organisation (town, country): Jülich, Germany
Annual operating costs (excl. investment costs) of the infrastructure (€): 3.150.000 €

1. Running Transnational Access Activities

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2028: Performance and characterization of two activated carbons for DMS and COS removal

One of the solutions that could contribute to decrease dependence from fossil fuels is represented by the exploitation of fuels from biomass through anaerobic digestion. The use of this renewable source determines a very low environmental impact but the resulting biogas is generally poor in energy content. For this reason, biogas can be exploited using a high efficiency conversion system, such as high temperature fuel cells, enhanced in a micro-cogeneration plant. Unfortunately, sulphur compounds, halogenated hydrocarbons and siloxanes can often be found in biogas. The main sulphur compounds present in biogas are hydrogen sulphide H2S (100-1000 ppm), mercaptanes (0-100 ppm) and traces of COS, CS2 and SO2. H2S represents one of the most harmful compounds for environment and working equipment. In fuel cells applications, H2S poisons both the reformer and the electrodes catalysts of the fuel cell, reacting with nickel; consequently, the biogas should be purified and H2S fraction reduced down to the tolerance limit (< 1 ppm) to allow its safe use. There are many clean up technologies to reduce sulphur levels, but not all can reach ultra-low sulphur for fuel cell applications. In this project, sulphur removal through adsorption systems is studied: the behaviour and adsorption capacity of selected adsorbent materials are investigated for DMS and COS removal.

Experimental activities started on May 2014 with a duration of six months, but at the time of writing this report, no final results were available.

Project 2045: Optimising Scandia stabilised Zirconia SOFC

Conventional Nickel-Yttria Stabilised Zirconia (Ni-YSZ) is the most developed and most commonly used anode because of its low cost and exceptional performance in H2 rich environments but under hydrocarbon operation, Ni-YSZ can deteriorate significantly due to low sulphur tolerances and carbon deposition. Developing SOFC systems that suppress coking and operate in lower temperature regimes improves system stability, lowers materials degradation. SOFCs based on Scandia-Stabilised Zirconia (ScSZ) are better suited than Yttria-Stablised Zirconia (YSZ) for use in low to intermediate temperature applications due to their higher conductivity values when compared against all of the suitable Zirconia dopants and the effect of Ceria in supporting carbon conversion. The project proposed here will support the work in Birmingham by:

1. offering calibration towards a well-established and experienced laboratory; this will allow better judgment of our own results on tailor made test rigs

2. offer the aspect of long-term operation we cannot currently perform on our own equipment ScSZ SOFC cells manufactured at UoB will be sent to JUELICH for 1000 to 3000 hours of testing under hydrogen and under methane internal reforming conditions. Supporting characterisation with leak testing will be helpful. Depending on the results, further experiments with contaminated fuel and under varying conditions supporting carbon formation will be conducted in order to gain supportive data on the performance of the UoB cells.

The start of work was envisioned for the last three month of 2014. Unfortunately the customer has not supplied us with any material or cell up to this date despite several requests from Jülich.

2. Description of installations provided

- **Durability testing of SOFC (DurSOFC):** The Solid Oxide Fuel Cell (SOFC) infrastructure at Jülich belongs to one of the largest SOFC research groups in the world, which possesses core research facilities in line with that scaling. The SOFC specific facilities (distributed across 5 main speciality institutes) are backed up by world class engineering, chemical analysis, computing and other relevant services from less involved campus institutes. Jülich produces a high number of scientific publications in the field – these results from the scale of the infrastructure and the number of relevant experts employed at Jülich. Significant competence (exceeding critical mass) is maintained in all relevant research areas from very fundamental materials development through to the design and construction of prototype pre-commercial systems. In addition to internal and external projects (German, EU and international), contract commercial research is carried on in the field on behalf of blue-chip companies such as BMW and ElringKlinger, but also SME's. New technology transfer is a core mission.
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3. Services offered

- **Durability testing of SOFC (DurSOFC):** The SOFC infrastructure has not engaged previously in any form of open access activities. Research services offered have been under contract to EU projects (including the coordination of Integrated Projects) and in other national and international collaborative projects, carrying out technology transfer with commercial partners, and in offering placements for students and guest scientists from universities and other organisations with which Jülich have come to formal or informal agreements to carry out mutually beneficial research activities. Cooperation is ongoing with partners from countries as varied as Brazil (COPPETEC/University of Rio de Janeiro), Russia (Institute of High Temperature Electrochemistry, Ekaterinburg), Ukraine (Institute of Problems in Materials Science, Kyiv), USA (ORNL), Bulgaria (UCTM, Sofia), France (SaintGobain, EdF and EIfER), etc. Many activities are undertaken with visitors from all countries and a significant number of employees and guests are non-German. Access to a world-leading supercomputer service via the SOFC infrastructure can be offered on a cost basis for advanced modelling applications in all project areas. The Jülich computing services institute is completely independent of the SOFC infrastructure. This institute offers generic computing services externally under its own scheme.

4. Description of work

Activity Plan

a) Jülich will provide SoA manufacturing facilities to the user to manufacture standardised test samples using the user's own cell materials. In some cases Jülich may produce the materials themselves according to the user's specification should the user be unable to make sufficient quantities. Materials other than the user's in the sample cells will be Jülich standard materials, these are well characterised. Standardisation of non-user materials is applied to ensure comparability of user results against fully characterised and benchmarked SoA cells produced internally at Jülich. All manufacturing technology/facilities of the ceramics institute (essentially every technique employed in this field) will be available in this activity, to aid ensuring that standardised cells can be successfully manufactured. Users may manufacture their own cells and eliminate this stage, but this is strongly discouraged as sample specifications for b) are strict to ensure eventual data comparability. This stage will involve most user presence.

b) User's will transfer to the electrochemical measurement institute and be involved in preliminary performance benchmarking of the standardised cells incorporating their materials. The long term testing samples will be mounted, the measurements started and the user will return home to wait for the completion of this testing element. The user will be informed of any significant changes in performance that occur suddenly and will receive periodic updates informing them of progress otherwise.

c) At the end of the long term test period, the user will return. Data will be analysed with an expert in data processing and durability issues. Performance loss (if any) will be calculated on a standard Jülich basis. The user will be instructed on best practise for doing this and on how best to present this data in publications, though the user has freedom in this. The user will be provided with all data collected in electronic and hardcopy form. All data will be prepared in such a way as to be of publishable quality. The user will then discuss the post-test analysis of the cells used in b) with durability study experts and a plan will be formulated to carry out chemical and physical analysis of the samples. Samples tested earlier for only a few days will be used for comparison purposes. All Jülich analytical facilities will be made available for this work, in each case the scientist carrying out the analysis type will be an SOFC expert in addition to having analysis type specific expertise.

Final results will be discussed between the user and Jülich staff freely (via email, phone etc.) with costed project time subsequent to the completion of the activities described above. Due to the different nature of the type of work, a different cost basis is used to account for the 24hr/365day capacity basis of long term testing facilities

Transnational Access @ PSI

Description of the infrastructure
Name of the infrastructure: SINQ, imaging stations NEUTRA and ICON
Location (town, country): Villigen, CH
Web site address: http://neutra.web.psi.ch
Legal name of organisation operating the infrastructure: Paul Scherrer Institut, PSI
Location of organisation (town, country):CH-5232 Villigen
Annual operating costs (excl. investment costs) of the infrastructure (€): 6.473.000 € (as of 2007)

1. Running Transnational Access Activities

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2008: Local water content distribution measurements with neutron imaging for CFD model validation of 50 cm2 PEMFCs

Status of the project: Finished

Technical installation used: SINQ, NEUTRA beam line, Fuel cell test bench

Project execution: The measurements were conducted from 23.7.2013 to 27.7.2013. Some parts of the measurement time (23.7 and part of 24.7) were needed to solve issues in the fuel cell brought by the user. The rest of the time was dedicated to perform measurements on one cell of 50 cm2, including automated overnight operation. Variation of parameters included changes of current density, operating gas (air or pure O2), and operation at different gas flow humidity for the anode and cathode sides. After discussion with the user, a new purging method to distinguish the water present in the anode channels from the water present in the cathode channels was tested and successfully applied.

User/user group: A single user (A. Iranzo) visited PSI for these measurements

Costs for the visit*:* CHF 1050.-

Amount of units used*:* 5 days

Dissemination activities*:* 3 publications were accepted by the International Journal of Hydrogen Energy.:

1. Iranzo, A., P. Boillat, and F. Rosa, International Journal of Hydrogen Energy, 2014. 39(13): p. 7089-7099. http://dx.doi.org/10.1016/j.ijhydene.2014.02.115
2. Iranzo, A., et al., International Journal of Hydrogen Energy, 2014. 39(28): p. 15687-15695. http://dx.doi.org/10.1016/j.ijhydene.2014.07.101
3. Iranzo, A. and P. Boillat, International Journal of Hydrogen Energy, 2014. 39(30): p. 17240-17245. http://dx.doi.org/10.1016/j.ijhydene.2014.08.042

A fourth publication by PSI on the purging method is in preparation and will be submitted to the

Journal of the Electrochemical Society before the end of 2014.

An image taken out of this project was used for the promotion flyer and for a poster at PSI describing neutron imaging of fuel cells.

Project 2014: Imaging Water Profile in Alkaline Exchange Membrane Fuel Cells

Status of the project: Finished

Technical installation used: SINQ, ICON beam line, Fuel cell test bench

Project execution: The measurements were conducted from 18.9.2013 to 22.9.2013. Three different large scale (250 cm2) cells were measured with the following schedule: 18.9.2013: setup of experiment / 19-20.9.2013 measurement of cell 1 / 21.9.2013 measurement of cell 2 / 22.9.2013 measurement of cell 3. The measurement of each cell included an activation period plus the measurements at different operating conditions. A purge method similar to the one first experimented in project 2008 was applied to intend distinguishing the water distribution between the different layers of the cells.

User/user group: Two users (S. Gottesfeld and M. Page) visited PSI for these measurements

Costs for the visit*:* The travel costs were not claimed yet by the users

Amount of units *used:* 5 days

Dissemination activities*:* The results obtained were presented at the Gordon Conference 2014 by S. Gottesfeld. A publication in a peer reviewed journal is under discussion.

Project 2015: Degradation effects of PEMFC in the edge region of the active cell area

Status of the project: Finished

Technical installation used: SINQ, ICON beam line, Fuel cell test bench

Project execution: The measurements were conducted from 17.12.2013 to 21.12.2013. Five different cells designs with an active area of 50 cm2 were tested during these measurements with approximately one day of testing for each cell. The parameters changed were the humidity of the anode and of the cathode, the cell temperature, the cell pressure and the applied current.

Besides imaging, advanced methods were applied in the frame of this project: firstly, the decompression purge method introduced in project 2008 was applied. This was partly successful to wip the water from the active area, but this method was not able to remove the water from the edge regions (outside of the active area) for some of the cell designs. Furthermore, an isotope exchange method (operating the cell with D2 instead of H2 for short periods of time) was applied to analyse the exchange processes within the cells.

User/user group: A single user (P. Stahl) visited PSI for these measurements

Costs for the visit*:* The travel costs were not claimed yet by the users

Amount of units used*:* 5 days

Dissemination activities*:* A paper will be submitted to the Journal of the Electrochemical Society before the end of 2014.

Project 2042: Water Profile Imaging in Alkaline Membrane Fuel Cells

Status of the project: Measurements finished, processing in progress

Technical installation used: SINQ, ICON beam line, Multicell setup, Fuel cell test bench

Project execution: The measurements were conducted from 3.8.2014 to 7.8.2014. Two different cell types were used: firstly, small scale alkaline membrane fuel cells (1 cm2) operated in differential mode using the PSI multicell setup. Secondly, a 14-cells stack of large scale (250 cm2) alkaline membrane fuel cells. The first two days were dedicated to measurements with the small scale differential cells, realizing the first ever *in plane* imaging of alkaline fuel cells. The third day (5.8.2014) was dedicated to the setup of the stack measurement, and the last two days (6.8.2014 and 7.8.2014) were dedicated to the measurements of the stack.

User/user group: A single user (M. Page) visited PSI for these measurements

Project 2043: Investigation of phenomena affecting the liquid water balance in a 50 cm2 PEFC

Status of the project: Measurements finished, processing in progress

Technical installation used: SINQ, NEUTRA beam line, Fuel cell test bench

Project execution: The measurements were conducted from 19.11.2014 to 23.11.2014. Although the measurements of two different cells were originally planned, technical issues in one of the cell appeared and only one cell could be measured. The first part of the beam time (19.11 and 20.11) was dedicated to setup of the test infrastructure as well as solving some leak issues with the cell. Subsequently, measurements were performed with variations of cell operation parameters (mostly humidity). The purge method introduced in project 2008 for the analysis of the difference between anode and cathode sides was applied again in this project.

User/user group: A single user (A. Iranzo) visited PSI for these measurements

Project 2044: Freezing behavior of PEFC in the outer perimeter of the active cell area

Status of the project: Measurements finished, processing in progress

Technical installation used: SINQ, ICON beam line

Project execution: The measurements were conducted from 25.7.2014 to 29.7.2014. A total of 3 cells with an active area of 50 cm2 were measured with the following schedule: 25.7.2014 measurement of first cell, 26.7.2014 measurement of second cell, 27-29.7.2014 extended measurements of 3rd cell. A recently developed method called “dual spectrum imaging” to identify differences between water in liquid and solid state was applied. Detailed analysis of the results is still in progress.

User/user group: A single user (P. Stahl) visited PSI for these measurements

Project 2055: Investigation of liquid water accumulation during oscillations in a PEFC with SINQ

Status of the project: Measurements to be performed in 2015

Technical installation used: SINQ, beam line to be chosen, Fuel cell test bench

Project execution: The user asked to perform the experiments next year in order to have enough time for preparing them. They will be conducted after SINQ restarts operation in April/May 2015.

User/user group: How many people will visit PSI is not determined yet

Project 2058: Investigation of the water distribution, coupled with current density and temperature mapping

Status of the project: Measurements to be performed in 2014

Technical installation used: SINQ, NEUTRA beam line

Project execution: The measurements are scheduled from 17.12.2014 to 19.12.2014.

2. Services offered

The neutron imaging and fuel cell test infrastructure at PSI allow imaging of fuel cells of different sizes in through plane (membrane perpendicular to the beam) and in plane (membrane parallel to the beam) configurations.

- **NEUTRA beam line:** The NEUTRA beam line was used for two access projects (2008 and 2043). In the so called through plane configuration (membrane perpendicular to the beam). The cells used in these user projects had an active area of 50 cm2. The NEUTRA beam line will be used as well for the user project 2058 schedules in December.
- **ICON beam line:** The ICON beam line was used for four access projects (2014, 2015, 2042, 2044). Two different measuring positions were used: the middle position with a middle sized beam for the cells in projects 2015 and 2044 having an active area of 50 cm2 and the read position with a larger beam for the cells in projects 2014 having an active area of 250 cm2. In the project 2042, both position were used: the middle position for high resolution in plane imaging of the multicell setup, and the rear position for imaging of 14-cells stacks.

A **mobile fuel cell test bench** is available at PSI for neutron imaging measurements of fuel cells

The mobile fuel cell testbench was used for all 6 projects conducted up to now. In projects 2008, 2015 and 2043, the standard configuration of the testbench was used. In projects 2014 and 2042, an extended version of for operating large cells (including for example a high current load with 360A capability). In project 2044, an extended version of the testbench for operating the cell at subzero temperatures was configured.

A **multicell setup** was recently developed, which allows the simultaneous in plane or through plane imaging of 6 small scale (1 cm^2 of active area) differential cells in the very same operating conditions.

This multicell setup was used in one single user project (2042) up to now.

Advanced methods.

Since the beginning of the projects, the following advanced methods (as compared to the standard and high resolution imaging described in the DOW) were proposed to the users:

1) Distinction of water in different layers of the cell by means of a special purging method. This method was first proposed in the project no 2008 and was since then applied is several different user projects (2014, 2015, 2043 and 2044).
2) Isotope exchange analysis. This allows to draw conclusions on the exchange processes in the cell by operating the cell on deuterium gas and/or with heavy water humidification. PSI has long term experience (Oberholzer, P. and P. Boillat, Journal of Physical Chemistry C, 2013. 117(39): p. 19945-19954. http://dx.doi.org/10.1021/Jp4045435; Boillat, P., et al., Electrochemistry Communications, 2008. 10(9): p. 1311-1314. http://dx.doi.org/10.1016/j.elecom.2008.06.016; Boillat, P., et al., Journal of Physics-Condensed Matter, 2011. 23(23). http://dx.doi.org/10.1088/0953-8984/23/23/234108) in isotope exchange experiments with neutron imaging and this experience was offered to the users in the frame of projects 2014 and 2015.

Dual spectrum measurements. The possibility of distinguishing between liquid water and ice using the differences of inelastic neutron scattering in the lowers range of the neutron energy spectrum was recently demonstrated at PSI (Biesdorf, J., et al., Physical Review Letters, 2014. 112(24). http://dx.doi.org/10.1103/Physrevlett.112.248301). This new method was put at the disposition of the user in the frame of project number 2044.

3. Description of work

The PSI Electrochemistry Laboratory (ECL) and the PSI Neutron Imaging and Activation Group (NIAG) propose the following support for collaboration project using the infrastructure described above:

- Identification of the most suited imaging setup for the desired purpose
- Verification of the compatibility of the fuel cells for neutron imaging, or indication of any necessary modifications
- Conduction of the experiments in collaboration with the visiting scientists or industrial partners
- Image processing of the obtained results
- For imaging with differential cells, the standard PSI cells can be used with specific materials (e.g. MEAs or gas diffusion layers)

Beam lines and detectors

Two beam lines are available for neutron imaging at PSI:

The NEUTRA beam line (http://www.psi.ch/sinq/neutra/neutra) have a thermal neutron beam with a flux of 0.75·107 n/cm2/s at the rear position and 1.5·107 n/cm2/s at the middle position. It is suitable for imaging up to very large cells as the beam at the rear position has a diameter of 40 cm and the largest detector option has a field of view of 35 x 38 cm2. Imaging of intermediate size cells with intermediate resolution is also possible.

The ICON beam lines has a cold neutron beam with a flux of 0.6·107 n/cm2/s and 2·107 n/cm2/s respectively at the read and middle positions when using the standard neutron source opening of 20 mm. A source opening selector wheel allows increasing the flux at the cost of having a more divergent beam (resulting in lower resolution due to the cell-detector distance). The ICON beam line also features additional options such as energy selective imaging using a neutron velocity selector and differential phase contrast imaging using a grating interferometer. The beam is smaller than at the NEUTRA beam line but still allows imaging of objects with a size of approx. 20 x 30 cm at the rear position.

The detector options cover a large range of fields of view, from a small field of view of 3 x 3 cm2 to the largest field of view of 35 x 38 cm2. The pixel and effective spatial resolution are dependent on the detector used. For imaging of objects with a large aspect ratio (such as fuel cells seen from the side), a series of tilted detectors are available, which increase the resolution specifically in one direction.

Fuel cell test infrastructure

A mobile fuel cell test bench is available for the measurements realized under the H2FC transnational access. The standard testbench configuration allows operation of small scale to middle size (e.g. 50 cm2) fuel cells. Different extension options are available to operate larger cells (e.g. electrical load up to 260A).

Additionally, a multi-cell setup was recently developed and is available for simultaneous operation and imaging of up to 6 small scale differential cells.

Advanced methods

In addition, the following advanced methods (as compared to the standard and high resolution imaging described in the original DOW) are available:

1) Distinction of water in different layers of the cell by means of a special purging method. The purging method consists in depressurizing successively the anode and cathode compartments to flush the water out of the flow channels, without affecting the water present in the gas diffusion layers or in the membrane. Analysis of the differences in the images before and after the purges allows to obtain separate images of the water present in the anode flow channels, and cathode flow channels. The remaining water after the purge is the water present in the porous media and membrane.

2) Isotope exchange analysis. This allows to draw conclusions on the exchange processes in the cell by operating the cell on deuterium gas and/or with heavy water humidification. PSI has long term experience (Oberholzer, P. and P. Boillat, Journal of Physical Chemistry C, 2013. 117(39): p. 19945-19954. http://dx.doi.org/10.1021/Jp4045435; Boillat, P., et al., Electrochemistry Communications, 2008. 10(9): p. 1311-1314. http://dx.doi.org/10.1016/j.elecom.2008.06.016; Boillat, P., et al., Journal of Physics-Condensed Matter, 2011. 23(23). http://dx.doi.org/10.1088/0953-8984/23/23/234108) in isotope exchange experiments with neutron imaging.

3) Dual spectrum measurements. The possibility of distinguishing between liquid water and ice using the differences of inelastic neutron scattering in the lowers range of the neutron energy spectrum was recently demonstrated at PSI (Biesdorf, J., et al., Physical Review Letters, 2014. 112(24). http://dx.doi.org/10.1103/Physrevlett.112.248301).

4) Double dead-end calibration. This method was developed in the frame of WP8 (JRA2.3.1) and consists in producing a determined quantity of water in a special flow configuration (double dead-end) ensuring that all produced water will stay in the cell, allowing quantitative comparison with the neutron imaging results and quantitative correction of the measurement results.

Transnational Access @ NCSRD

Description of the infrastructure
Name of the infrastructure: HYSORB
Location (town, country): Athens, Greece
Web site address: www.demokritos.gr
Legal name of organisation operating the infrastructure: National Centre for Scientific Research "Demokritos"
Location of organisation (town, country): Athens, Greece
Annual operating costs (excl. investment costs) of the infrastructure (€): 143,080 € (as of 2009)

1. Running Transnational Access Activities

By the end of October 2014, the HYSORB facility offered/is offering experimental time to 4 user user projects, as listed below.
All experiments so far were quite lengthy due to the large number of samples studied as well as the time consuming nature of mainly the gas sorption measurements required (including the absolutely necessary pre-treatment of the samples). Besides it was already foreseen that 1 week is the minimum amount of meaningful access for HYSORB facility (actually corresponding to the processing of one sample).

The main objectives and obtained results of the completed so far experiments are summarized per project as follows.

Project 2002: Investigation of the hydrogen storage and thermal properties of (La-Ce)Ni5 type alloys and their composites.

The main purpose of the experiments was to investigate the effect of the partial substitution of La with Ce on the crystal structure and the final hydrogen absorption/desorption properties of new (La-Ce)Ni5 type compounds. H2 absorption and desorption experiments at different temperatures were performed on 8 in total samples of La1-xCexNi5 type compounds with varying Ce content (x = 0-0.8), using a PCTPro-2000 automatic volumetric system. H2 absorption/desorption isotherms were recorded at different temperatures ranging from 20oC to 90oC. X-ray powder diffraction was also performed before and after hydrogenation, to investigate the microstructural changes of the samples. Some preliminary in-situ calorimetry/H2 sorption experiments were performed but it was decided for technical reasons to conduct a complete series of such measurements in a future experiment. The study of the structural and H2 sorption properties of the examined compounds provided information about the effect of the partial substitution of La by Ce in the crystal structure of LaNi5. It was seen that although the characteristic microstructure of the original LaNi5 alloy was maintained, the incorporation of Ce led to the contraction of the unit cell. This geometrical change might be responsible for the alteration of the H2 storage properties of the respective systems.

Project 2003: Hydrogen storage measurements in oxidised and metal decorated single-wall carbon nanohorns.

The proposers showed previously that Single Wall Carbon Nanohorns (SWNHs) interact more strongly with the SWNHs compared to carbon nanotubes, suggesting that nanohorns may offer significantly better prospects as lightweight media for H_2 storage applications. For a more complete of the H_2 storage properties of the developed materials it was necessary to perform a systematic series of H_2 adsorption/desorption measurements at different temperature and pressure conditions. The samples were characterized with regard to their structural/morphological properties by different methods (FTIR, TGA, XRPD, SEM), as well as their H_2 sorption performance at different temperatures (77 K, 298 K) and pressures (1-120 bar). N_2 adsorption–desorption isotherms obtained at 77 K were used to determine key porous properties (surface area, pore volume values). The H_2 storage capacity of the materials was determined at 77 and 298 K up to 120 bar, using both gravimetric and volumetric methods. Overall the carbon samples showed very low hydrogen uptake (less than 0.1 wt%) at room temperature that increased practically linearly with pressure (Henry-like behaviour), while no hysteresis was observed during desorption..

Project 2013: Carbon-based nanostructures for hydrogen storage.

The main focus of the specific project was the experimental study of an extensive series of sorbents with different structural and porous characteristics, in an attempt to address the main classes of porous materials (carbon nanostructures, framework materials etc.) currently receiving attention as potential H_2 stores. In this respect, both commercially available (carbon nanotubes, layered graphene, carbon black) and newly synthesized (carbon aerogels, exfoliated graphene oxide, metal-organic frameworks etc.) nanomaterials were systematically examined. The textural properties of the materials were determined by N_2 adsorption at 77K; important parameters were extracted such as the BET specific surface area, the total pore volume and the pore size distribution. Additional methods such as X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Infrared Spectroscopy (IR), Thermogravimetric Analysis (TGA), were also employed in order to elucidate further their structural features. The H_2 sorption behaviour of each material was studied by systematic adsorption/desorption measurements at different pressures (0-20bar) and temperatures (77K-298K) using specialized low and high pressure volumetric systems.

Project 2064: MOFs as dihydrogen containers.

The proposers are working on the design and development of porous MOFs that can selectively adsorb large H_2 quantities. To date, they have prepared 5 different MOFs, for the accurate characterisation of which it is necessary to perform a systematic series of H_2 adsorption/desorption measurements at least at cryogenic temperatures and pressures ranging between 1-120 bar. In addition a comparative study of their sorption capacity with respect to other technologically relevant gases (e.g. CO_2, CH_4, N_2) also needs to be carried out. The experimental work for this specific project started in the second half of October 2014 and is expected to be completed by early December 2014. The overall methodology is based on a wide range of measurements, such as: TGA measurements on all samples to verify complete removal of solvents, N_2 adsorption/desorption measurements at 77K for the determination of the pore properties (specific surface area, pore size/volume etc.) of all samples, low pressure (up to 1 bar) volumetric H_2 sorption measurements at 77 K, high pressure (up to 120 bar) volumetric H_2 sorption measurements at 77 K etc.

In the case of projects 2003, 2003, 2064 the samples were sent to HYSORB and the experiments were carried out without the presence of the users (however, there was a continuous communication/feedback throughout). On the other hand, the experiments of project 2013 comprised a major part of the MSc thesis of Mr. N. Kostoglou (post-graduate student in the Mechanical and Manufacturing Engineering Department, University of Cyprus, Cyprus). As such Mr. N. Kostoglou visited (at own expenses) repeatedly the HYSORB facility and attended/took part in (after extensive training) an extensive series of the respective measurements.
Although all experiments proved to be time consuming and laborious, the measurements were in principle concluded without major technical difficulties. It was realised however that in certain cases (e.g. Projects 2002, 2003) additional measurements with other methods and/or samples are necessary to produce publishable results. In contrast, Project 2013 had a key contribution to the completion of an MSc thesis and the generation of interesting scientific results

The following dissemination activities took place so far:

- Nikolaos Kostoglou, 'Solid-state Hydrogen Storage by Physical Adsorption in Nanoporous

Materials“, MSc Thesis, Department of Mechanical & Manufacturing Engineering, University of Cyprus, May 2014.

- N.K. Kostoglou, C. Rebholz, V. Tzitzios, C. Tampaxis, T.A. Steriotis, K. Giannakopoulos, G.C. Charalambopoulou, K. Polychronopoulou, Y. Li, K. Liao, “Hydrogen storage capacity of different nanoporous carbon adsorbents”, 30th Panhellenic Conference on Solid-State Physics and Materials Science Heraklion, Crete, 21-24 September, 2014.
- N. Kostoglou, V. Tzitzios, Ch. Tampaxis, G. Charalambopoulou, Th. Steriotis, K. Giannakopoulos, A. Kontos, Y. Li, K. Liao, K. Polychronopoulou, C. Rebholz, “Synthesis, characterization and hydrogen storage capacity of nanoporous graphene-based adsorbents”, AVS 61st International Symposium & Exhibition, Baltimore-USA, 9-14 November 2014.
- N. Kostoglou, V. Tzitzios, Ch. Tampaxis, G. Charalambopoulou, Th. Steriotis, K. Giannakopoulos, A. Kontos, Y. Li, K. Liao, K. Polychronopoulou, Claus Rebholz, Synthesis, characterization and hydrogen storage capacity of nanoporous graphene-based adsorbents, International Journal of Hydrogen Energy, submitted

The HYSORB technical experts are currently discussing the possibility of at least three more user projects with groups from the Netherlands, the Czech Republic as well as Austria. These experiments mainly concern the study of challenging nanostructured materials that have not been studied extensively for hydrogen storage. When considering the realisation of these new projects as well, it is expected to match (or even slightly exceed) the originally foreseen time for transnational access to HYSORB (210 days).

2. Description of installations provided

- **HYSORB:** HYSORB is the hydrogen sorption laboratory of the National Centre for Scientific Research “Demokritos”. Its facilities congregates a rarely met collection of state-of-the-art, top-class and mainly complementary instrumentation that allows accurate H2 storage measurements in well controlled sample environment at different pressure/temperature conditions (vacuum - 200 bar, 9 - 600 K) and scales (material quantities from mg to bed scale), predominantly for the study of physisorption/weak chemisorption phenomena in sorbents. More specifically, a wide range of volumetric, gravimetric, calorimetric and spectroscopic techniques are offered including: an Intelligent Gravimetric Analyser (IGA, 77-600 K, vac-20 bar), a magnetically suspended Rubotherm balance (vac-200 bar, 77-1000 K), a commercial PCT volumetric system (0-170 bar, 77-600 K) also equipped with a micro-dosing module for accurate PCT measurements on very small sample quantities, two low pressure (<1 bar) but extremely high resolution volumetric apparatuses, a customised Thermal Desorption Spectroscopy coupled with MS rig (9-600 K), a Setaram C80 gas tight Calvet Calorimeter (1-350 bar, ambient–600K), a TGA/DSC/MS system (90-1500 K).

3. Services offered

- **HYSORB:** The available equipment at HYSORB experimental facility comprises a rarely met toolbox and as a result the laboratory is now operating as a national infrastructure mainly for high pressure hydrogen measurements (continuous testing/screening of materials developed also by other laboratories). Moreover, the group has also taken part in interlaboratory comparisons (bilateral collaborations with e.g. JRC-Petten, SwRI-USA, NREL-USA, University of Birmingham-UK; participation to round robin tests of FP6 projects NESSHY and HYSIC) for the independent assessment of the performance of new materials, but also for the formulation of common measurement protocols. In addition, services related to the thermal design (e.g. heat management) of solid storage systems (tanks) along with much desired optimisation studies for the efficient development of storage tanks have been provided (with the aid of the gPROMS based simulation tool) in the framework of several projects (e.g. FP5-HYSTORY, FP6-STORHY, FP6-NESSHY).

4. Description of work

HYSORB facilities are already known within the hydrogen scientific community through the existing projects, international collaborations and networking activities. Outreaching to potential external users will be further developed through personal contacts and dissemination routes (hydrogen conferences, workshops, exhibitions) also in conjuction with networking tasks N4.

Interested users will submit their requests/applications on a quarterly basis. The final selection and scheduling of the proposed measurements will be based on the operational program and capabilities of the HYSORB laboratory at the time. For the implementation of the accepted experiments the presence of the external users on site is not mandatory. The necessary sample quantities can be sent to HYSORB responsible staff given that sufficient background information and detailed instructions for handling and testing conditions are provided. In the opposite case, the external users will visit the facility at an agreed time and will be trained on the basic experimental and safety practices prior the start of the actual measuring time. Full support of the experienced scientific HYSORB staff will be provided throughout the duration of the experiment. After its completion the full set of obtained data (in raw and/or preliminary processed form) will be delivered to the user (backups will be also available). Users will be also supported if needed for data processing and analysis but also for the interpretation and publication of the results.

Transnational Access @ UP

Description of the infrastructure
Name of the infrastructure: **The Fuel Cell and Hydrogen Laboratory**
Location (town, country): The Lab comprises two installations at the following different locations: 1. FCLab (Fuel Cell Laboratory) at the Department of Industrial Engineering, located in Via G. Duranti, 67 – 06125 Perugia, Italy; 2. MCLab (Material Characterization Laboratory) at the Department of Physics, located in Via A. Pascoli, Perugia, Italy.
Web site address: www.unipg.it
Legal name of organisation operating the infrastructure: Università degli Studi di Perugia
Location of organisation (town, country): P.zza Università n.1, 06125 Perugia (Italy)
Annual operating costs (excl. investment costs) of the infrastructure (€): 300.000,00

1. **Running Transnational Access Activities**

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2011: Role of hydrogen bounding in proton conductivity of Nafion membranes

Installation: X-ray

Finished project;

Project 2049: Innovative catalysts for the steam reforming of renewable alcohols

Installation: X-ray

Project ongoing;

Project 2020: Molten Carbonate Fuel Cell: Identification of the performances for modelling purposes

Installation: MCFC

Finished project;

Project 2035: Analysis of CO2 separation with MCFCs operating on simulated exhaust gases from biogas engines

Installation: MCFC

Project ongoing;

Project 2025: Solid oxide fuel cell experimental analysis with different hydrocarbons

Installation: SOFC

Finished project

Project 2036: Operate SOFC with Bio-Gas

Installation: SOFC

Project ongoing;

Project 2053: Multisource multiproduct energy systems with multifunctional fuel cells

Installation: SOFC

Project ongoing;

Project 2024: Performance and characterization of two activated carbons fo biogas treatment applications

Installation: CDS

Finished project;

Project 2056: Evaluation of a novel absorbent for anaerobic gas desulfurization

Installation: CDS

Project ongoing;

2. Description of installations provided

As mentioned here above, the Fuel Cell and Hydrogen Laboratory of the University of Perugia comprises two installations at the following different locations:

1. FCLab (Fuel Cell Laboratory) at the Department of Industrial Engineering;
2. MCLab (Material Characterization Laboratory) at the Department of Physics.

The installations we will offer to external users within H2Fc project are:
a) **N. 1 test bench** for experimental activity on small scale reforming devices, concerning both conventional reforming and Sorption-Enhanced steam methane reforming (test of CO_2-sorbent materials and catalysts and characterization of the effect of reforming operative conditions).
b) **N. 1 test bench** (FCpowers of 300 W) for SOFC full equipped for tests of degradation effects, performance evaluation and life cycle characterization of FC in multi fuels feeding case;
c) **N. 1 test bench** (FCpowers of 300 W) for MCFC full equipped for tests of degradation effects, performance evaluation and life cycle characterization of FC in multi fuels feeding case.

The installations we will offer to external users within H2Fc project are:

- N.1 Small and wide angle x-ray diffractometers for powders and crystalline specimens. Two x-ray generators (3 kW power, 0 - 60 kV voltage) are available. Characterization of cell materials in different working conditions is routinely possible. Both operating temperature and other parameters like pressure can be controlled during the measurements. Compton profile analysis is also available with excitation energies from 8 keV (Cu anode) up to 60 keV (W anode).

- **N. 1 Neutron CRG Low spectrometer angle Brillouin** to access the kinematic region 0-10 THz, 2 – 20 nm^{-1}.

3. Services offered

The main research areas of Unipg Lab are: performance analysis of high temperature FCs; integrated systems based on FCs and BoP analysis; carbon capture sequestration CCS and hydrogen production also through Sorption-Enhanced Steam Methane Reforming, which allows contextual CO2 separation through adsorption from renewable sources (biomass-derived syngas and gas mixtures mainly constituted from CH4 and CO2 such as land-fill gas and digester gas). Here below the detail of the above mentioned areas:

1) Testing on MCFC and SOFC: performance analysis and evaluation with alternative fuels usage, pollutants effects assessment and durability analysis;
2) Testing on fuel processing matters both on desulphurization and reforming, relative to materials (sorbent/catalyst and CO2 acceptors) performance and process operative conditions;
3) Analysis and optimization through simulation of FCs integrated and hybrid (FCs coupling with gas turbine, internal combustion engine, ...) systems and BoP components, both as system integration and components design ;
4) Analysis and optimization through simulation of conventional SMR and SE-SMR devices, both as system integration and components design;
5) Stationary numerical simulations: numerical codes development concerning FCs, FC integrated and hybrid systems at regimen and partial loads, hydrogen production systems;
6) Dynamic simulation and control strategy: development of dynamic models of integrated FC systems both for stationary and vehicular applications (dynamic analysis of FC/battery pack power system) aiming at the dynamic analysis and control algorithm development;
7) CFD numerical simulation: development of CFD models of single components of FC integrated systems and H2-production plants, with implementation of the kinetic of adsorption process (desulphurizer) and the chemical kinetics of reforming and CO2 adsorption in a solid phase (SE-SMR reactor) aiming at the component geometry optimization. Development of model for H2 release in indoor ambient aiming at the design of preventing means such as hydrogen leak detection and isolation;
8) Exergetic analysis, cost, environmental and reliability analysis (ex. LCA – Life-Cycle-Assessment). Such an analysis is also focused on the optimal H2 production system (from an energetic and exergetic point of view) in function of the fleet consistency;
9) The MCLab at the Department of Physics is mostly involved in material and device characterization activity to be carried out before and after the operational tests. This is achieved by using X-ray and neutron scattering methods that have already proved to be very helpful in elucidating the membrane structure, distribution of catalysts and understanding proton transport in fuel cells. Neutron radiography and tomography could be applied to visualise processes like water transport in the channels of the flow field and in the pores of the gas diffusion layers in operating fuel cells and stacks. Small angle neutron scattering (SANS) would yield the structure of ionic clusters in membranes, and reveals their anisotropy and swelling in water. The evolution of water in the cathode of a fuel cell during operation and consequent swelling or changes in the membrane can be visualized. X-ray scattering techniques would provide information on the distribution of different inorganic components (inorganic additives, catalysts) in the membrane. Increasing the efficiency and affordability of fuel cells requires a better understanding of the molecular-level processes involved in oxidation and reduction at electrodes, catalytic processes, and ion and proton transport, as well as the development of polymer electrolyte-membranes and solid oxide electrolytes. The performance of these components involves several sizes (nm to μm) and time scales which are readily accessible to X-ray and neutron scattering techniques.

As the Lab guarantees an high scientific and research quality level, it normally hosts, within thesis / master development and exchange programs purpose, students and researchers coming from all over the world. A range of at least four students/researchers per year are welcomed at Fuel Cell and Hydrogen Lab premises.

Thanks to several scientific publications and activities (participation at the most important workshops and conferences, meetings, etc.), the membership of N.ERGHY AISBL as well as the organization of the “European Fuel Cell Technology & Applications – Piero Lunghi Conference”, the Lab is really active in the international Fuel Cell and Hydrogen context.

Transnational Access @ ENEA

Description of the infrastructure
Name of the infrastructure: **The Fuel Cell and Hydrogen Laboratory**
Location (town, country): ENEA Casaccia Research Centre Rome - Italy.
Web site address: www.enea.it
Legal name of organisation operating the infrastructure: ENEA, Italian National Agency for new technologies and the sustainable economic development
Location of organisation (town, country): Rom Italy
Annual operating costs (excl. investment costs) of the infrastructure (€): 1.220.000 €

1. **Running Transnational Access Activities**

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2033: NTN Composite Interconnect Testing

Status: On-going: Tests are longterm (500 h each, 6 foreseen)

Facility: FC Test Lab

Objective: Benchmarking and long-term dual-atmosphere testing of samples of a novel interconnect material (coated with different cathode materials), including material characterization, in-situ measurement of resistance, post-test analysis to evaluate robustness and performance

Project 2057: Online monitoring of biogas composition by a new laser spectroscopic instrument

Status: On-going: Visits foreseen for March 2015

Facility: FC Test Lab

Objective: Extended field test of newly developed optical trace gas analysis systems at a real-world biogas/FC installation: ENEA will make their biogas digester available and prepare it for adequate cross-referencing of the new instruments with alternative trace gas analysis equipment

Project 2065: Performance and characterization of two activated carbons for DMS and COS removal

Status: On-going: delays due to slow delivery of calibrated technical gas

Facility: FC Test Lab

Objective: the behaviour and adsorption capacity of selected adsorbent materials are investigated for DMS and COS removal from biogas-like matrices through dedicated adsorption tests.

Project 2068: Investigation to the feasibility of MCFC as CCS technology

Status: Waiting for go-ahead from User Office

Facility: FC Test Lab

Objective: Investigate the feasibility of MCFC as CCS technology through extensive experimental campaigns focused on the evaluation of the effects of SO_2 in the cathode stream on cell performance including the integrated reformer catalysts.

Project 2069: Effects of SO_2 on MCFC

Status: go-ahead from User Office received in December 2014

Facility: FC Test Lab

Objective: Experimental study of the effects of SO_2 when fed to MCFC cathode, so that effects of contaminated oxidant and fuel can be investigated and numerically simulated, as SO_2 in the oxidant transfers thought the electrolyte from the cathode to the anode to form H_2S in the fuel.

2. Description of installations provided

- **FC Test Lab:** The FC testing laboratory consists of four laboratories: two of them consist of single cell and stack test benches for performance evaluation and degradation mechanism investigation of MCFC and SOFC. The third laboratory is dedicated to chemical-physical characterization of cell and stack components both pre and post-test. Finally, akin to the labs on fuel cell characterization is the laboratory for gas conditioning prior to fuel cell feeding. Here, a biogas pilot plant and several stations for the upgrading of biogas to fuel cell inlet conditions are present. The facilities are offered for field testing of clean-up materials and techniques and gas analysis techniques. The access to these 4 laboratories will allow the user to the complete characterization of systems based either on MCFC or on SOFC taking into account the specification needed for a peculiar application. For the present proposal, tests can be conducted to test performance and endurance of supplied cells and stacks in a number of applications, including stationary, micro-residential and APU - Auxiliary power units on board cars, buses, trucks, ships, caravans, boats… (SOFC, MCFC). Particular attention is devoted to applications using alternative fuels such as biogas and biosyngas, which is why the highly equipped fuel gas upgrading facilities are part of the infrastructure offered, yielding insight as to the monitoring of compositional variations of biofuels, pollutant control and retention, fuel upgrading and system integration and optimization.

3. Services offered

The access to these 4 laboratories will allow the user to the complete characterization of systems based either on MCFC or on SOFC taking into account the specification needed for a peculiar application. For the present proposal, tests can be conducted to test performance and endurance of supplied cells and stacks in a number of applications, including stationary, micro-residential and APU - Auxiliary power units on board cars, buses, trucks, ships, caravans, boats… (SOFC, MCFC). Particular attention is devoted to applications using alternative fuels such as biogas and biosyngas, which is why the highly equipped fuel gas upgrading facilities are part of the infrastructure offered, yielding insight as to the monitoring of compositional variations of biofuels, pollutant control and retention, fuel upgrading and system integration and optimization.

Transnational Access @ Tecnalia

Name of the infrastructure: **HyMAT**
Location (town, country): **San Sebastian, Spain**
Web site address: **www.tecnalia.com**
Legal name of organisation operating the infrastructure: **Fundacion TECNALIA**
Location of organisation (town, country): **San Sebastian, Spain**
Annual operating costs (excl. investment costs) of the infrastructure (€): 650.000 €

1. **Running Transnational Access Activities**

So far, 2 accesses were required. One of them is finished and closed (access 2016) and second is about to be closed (access 2017).

Project 2016: Hydrogen embrittlement of quenched and tempered steels

The main aim of the project was to carry out slow strain rate tests (SSRT) under different hydrogen charging conditions (neutral environment, sea water with cathodic protection and NACE solution A) using the facility offered by Tecnalia within the H2FC European Project, on high strength quenched and tempered low-alloy steels manufactured by Dalmine S.p.A., in order to appraisal its sensitivity to hydrogen embrittlement (HE). The resuls show a clear difference between inert condition (N_2 gas), were there is no sign of embrittlement and all the other conditions where evidences of HE were observed in terms of reduction of area, lower failure time and presence of secondary cracks. Nevertheless, there is a difference between the environments generating HE. The environment saturated in H_2S is the most aggressive for both references. The Ni enriched steel shows higher HE resistance compared to base steel under cathodic polarization conditions. However, in H_2S environment, beside the fact that there is an important degradation of the properties due the HE, the situation is opposite, with base steel showing better behavior compared to Ni enriched steel.

The project is finished and the final report was submitted online.

The installation used during this project were the corrosion laboratory of Tecnalia and the SSRT (slow strain rate test) device.

Project 2017: Identification of most resistant steel to corrosion in hydrogen present environments. New alloys comparison

The project is consisting in testing the sulphide stress cracking sensitivity of steel alloys following the standard Nace TM0284

The project is still ongoing. At the date of December 2014. The experimental part just finished and the data analysis is almost finished. We expect the redation of the final user report for end of january 2015 at maximum.

The project was extremelly delayed by time taken by Grerdau to provide the experimental samples (the experiment with the first material were finished in 2013 when and then we had to wait for mid 2014 to have the second alloy and fall 2014to have the third one.

2. **Description of installations provided**

The **Hy**drogen-**MAT**erials testing activity HyMAT provided by Tecnalia allows accessing to 3 kinds of installation:

- An HIC (hydrogen induced crackin) facility which consist in a speccific laboratory area with specific glassware equipment allowing to work safely with H_2S. The test is usually done following the standard NACE TM0284
- A SCC (stress corrosion cracking) faclity wihich consist in a set of 6 dynamomatric rings with glass cells used to apply a constant tansile load on the coupon. While it is immersed in an agressive environment. With our facility, it possible to have an hydrogen load using H_2S or cathodic polarization. The test is usually done following the standard NACE TM0177.
- SSRT equiment. The SSRT consist in a tensile machine working by applying a slow strain rate to the tensile coupon, associated with an Hastelloy autoclave where it is possibl to put the sample in contact with an agressive environment. The hastelloy autoclave alloy to use very agressive anvironment (very high corrosion resistance) as well as working in temperature (up to 300ºC) and pressure (up to 280 atm.). It is possible to exchange the autoclave by an electrochemical cell to an hydrogen load by cathodic polarization.

3. Services offered

At the HyMAT infrastructure it is possible to investigate the behaviour of materials in hydrogen environments. The influence of hydrogen in materials properties can be evaluated. The sensitivity of materials to specific phenomena of properties degradation as hydrogen embrittlement can be assessed. A team formed by some twelve people working in several laboratories working in mechanical, corrosion, chemical and microstructural characterization is offering the service of this infrastructure. HyMAT is actually used in several industry co-financed projects for diverse applications.

- Modality of access under this proposal

Within the framework of the project, open access will be provided to the HyMAT installation for maximum 10 days per year with minimal time periods of a week. In a general way the potential user will provide the information and the asked study will be performed by the staff of HyMAT. In the case of a highly experienced service demander, the user could perform part of the work helped by HyMAT team.

- Support offered under this proposal:

The HyMAT team will help and support materials issues for different hydrogen applications. General aspects as the selection of materials for applications in hydrogen environments and more particular ones as new ways of obtaining materials and components for Fuel Cells such as Membrane Electrode Assembly (MEA) obtained by modification or improvement of polymer properties by plasma polymerisation and the optimisation of the catalyst deposition by PVD Magnetron Sputtering Technologies. This can be done in a laboratory at TECNALIA that is actually working in the field of the development of materials for fuel cell and electrolysis cell.

4. Description of work

a) After a first contact with the user (though the contact done during the promotion of the facility or after a

b) The material is provided by access user by regular mail. It is usually given in its “final from” (e.g. section of tubes or pipes, ingots …”. Then the samples are machined in the workshop of Tecnalia in order to prepare specific coupons for the test chosen by the users (prismatic coupons for HIC test or tensile coupon for the SCC and SSRT tests).

c) The tests are running by Tecnalia technicians under the condition defined in the proposal.

d) The compilation of the results obtained and the data analysis is done by Tecnalia researchers and sent to the access users.

e) The final report is written by the users in collaboration with Tecnalia researchers and submitted to H2FC by the users.

Transnational Access @ NPL

Description of the infrastructure
Name of the infrastructure: Test facilities for characterisation of single cell PEFCs, electrochemical characterisation and hydrogen purity analysis by gas chromatography
Location (town, country): NPL, Teddington, United Kingdom
Web site address: www.npl.co.uk
Legal name of organisation operating the infrastructure: NPL Management Ltd
Location of organisation (town, country): Teddington, United Kingdom
Annual operating costs (excl. investment costs) of the infrastructure (€): no figure given

1. Running Transnational Access Activities

A short summary of the proposal and the work performed so far is given in the following paragraphs.

Project 2039: Study of electrosprayed deposited CCMs for PEMFC

NPL has had one access visit (from CIEMAT, Madrid) in 2014. This was the first project carried out at NPL under the H2FC access activities. The project is ongoing and scheduled for completion in January 2015.This project comprised a systematic study of the performance of catalyst coated membranes (CCM) prepared using an electrospray technique, which is based on the deposition of a solid material in a suspension under the influence of an intense electric field. It has been used by Dr Chaparro's group at CIEMAT for several years for the preparation of catalyst layers for electrodes and CCMs of polymer electrolyte membrane fuel cells (PEMFCs). The advantages of CCMs prepared by this method are larger specific area of the catalyst, improved interaction of the ionomer with the platinum catalyst, and optimal porosity and mass transport properties of the layers.

A series of electrosprayed CCMs were prepared by CIEMAT with variable Pt/C ratio in the cathode, maintaining the same catalyst load (0.25 mg•cm-2) and ionomer content (15 wt%). A standard electrode was used on the anode in order to compare cathode performance. Testing of the electrosprayed CCMs was carried out at NPL and included I-V curves, electroactive area measurements, startup/shutdown cycles and localised potential mapping. The electrosprayed CCMs showed performance comparable to that of a state-of the-art carbon-supported Pt electrode and exhibited good stability during start-up/shut-down cycling. The experimental work is now almost complete and preparation of the report will commence shortly.

Dr Chaparro visited NPL for the first week of the project to witness the test setup and discuss the optimum protocol for conducting the tests. Costs of his visit were claimed from the H2FC project.

It is planned to disseminate the project outputs via a joint publication in a peer-reviewed journal. Dr Chaparro will present the results of the project at the H2FC Technical School in Crete in June 2015. A poster will also be presented at the 9th International Symposium Hydrogen and Energy in

Emmentem, Switzerland in January 2015.

2. Description of installations provided

NPL1: Instrumented single cell PEFC test stations.

NPL has a dedicated PEFC research laboratory equipped with a range of novel in situ measurement techniques, with the principal focus on studying fuel cell degradation modes such as startup/shutdown and cell reversal. The facility contains two highly instrumented Hydrogenics single cell test stations, supported by a range of material and electrochemical characterisation techniques. Available techniques include unique capability for in situ measurement of relative humidity in PEFC flowfield channels and localised current density measurement using a segmented electrode. Ground-breaking in situ reference electrode capability will be added during this project.

Note that the following two installations (NPL2 and NPL3) were removed from the H2FC access list following the mid-term review due to lack of demand for their use.

NPL2: Electrochemical techniques for characterisation of catalyst performance.

- GC – mass detection.
- GC – thermal conductivity detection
- GC – flame ionisation detection
- GC – sulphur chemiluminescence detection
- GC – pulsed helium discharge ionisation detection.

The work we have been focusing on in this area has been on identifying key impurities at the levels specified in the latest drafts (or published versions of) ISO 14687-2 and 14687-3 on hydrogen purity. Resolution will be improved to sub ppm levels during project.

NPL3: Electrochemical techniques for characterisation of catalyst performance

3. Services offered

NPL has extensive facilities for electrochemical characterisation of fuel cell catalysts, including state of the art scanning electrochemical microscopy (SECM) and rotating disk electrode (RDE). SECM enables the characterisation of electrocatalyst materials through mapping of surface reactivity under relevant aqueous environments. Conventionally a microelectrode probe is scanned at a fixed distance from the substrate and electrolytic processes are driven and monitored locally by the probe to detect spatial variations in catalyst behaviour and activity. In the simplest case the activity of a catalyst sample towards the hydrogen oxidation reaction can be probed in acidic electrolytes, but the experiment can be tailored to a range of reactive systems (e.g. oxygen reduction reaction) and conditions (e.g. pH, solvent medium). Spatial resolution is typically of the order of microns across scanning areas as large as several millimetres. Information about electrochemical kinetics is also obtainable through probe approach curves at points of interest. RDE experiments can be undertaken to gain kinetic information about electrocatalytic processes by lifting mass transport limitations. Typically catalyst particles are immobilized onto a carbon RDE substrate and electrocatalytic activity with respect to the hydrogen oxidation and oxygen reduction reactions is measured through linear sweep and potential step measurements under controlled conditions.

Transnational Access @ EMPA

Description of the infrastructure
Name of the infrastructure: *Hydrogen and Energy*
Location (town, country): Dübendorf, Switzerland
Web site address: *www.empa.ch/h2e*
Legal name of organisation operating the infrastructure: *Eidgenössische Materialprüfungs- und Forschungsanstalt (EMPA)*
Location of organisation (town, country): 8600 Dübendorf, Switzerland
Annual operating costs (excl. investment costs) of the infrastructure (€): 80'000,- €

1. Running Transnational Access Activities

A short summary of the proposal and the work performed so far is given in the following paragraphs All of them are related to Lasy facility.

Project 2001: Raman spectroscopy on MgHx nanoparticles produced by spark discharge generation

The main objective for these measurements was to understand the binding of hydrogen to magnesium. This study could provide some insight in the mechanism of the hydrogenation and dehydrogenation of the samples. This will allow us to better tailor the materials we produce to be most efficient with hydrogen sorption reactions.

A series of six samples of MgHx synthesized in different mixtures of argon and hydrogen plus two magnesium hydride reference samples have been used to measure Raman and infra-red spectra. The Raman spectra could only be measured for the commercial MgH2 reference sample. The nanosized samples did not show any signal above the noise level. Dr. Borgshulte suggested that cooling the sample (~230K) during measurement will increase the signal. Even at low temperature the signal was very low, practically indistinguishable from the background level. However, during the cooling the window of the sample holder became very brittle and it broke during the removal of the sample. In parallel to the Raman measurements, Infrared measurements were successfully performed. The results obtained show very interesting effects due to the atmosphere in which the material is synthesized.

Though the Raman measurements did not provide any useful data, the infrared measurements show very interesting effects in the samples. The absence of an-OH band in the structure is a very encouraging result that the samples do not contain Mg(OH)2. For the samples that in XRD patterns are crystalline, the normal vibration spectrum of MgH2 is found while for the sample that appears to be amorphous in XRD, different bands are measured. The presence of vibration bands is an indication of a not completely random structure. We suspect it is an intermediate state between Mg metal and the hydride. The results obtained will be included in a manuscript of a paper that is now being written.

Raman measurements did not show any signal for the nanostructured MgH2 as we initially expected. The low temperature measurements (230K) have led to the breaking of the air tight sample holder.

Project 2018: Effects of Zirconium and Polytetrafluoroethylene on the hydrogen sensing

properties of Yttrium-Palladium based hydrogen indicator

The objective of the measurements was to investigate why the addition of small amounts of Zr to an Y thin film resulted in a significant change in its hydrogen sorption properties, and the addition of sputtered PTFE on Pd-based thin film catalysts led to a profound increase in the hydrogenation and dehydrogenation kinetics. For this we needed to do in-situ Raman and Infrared (IR) measurements to detect possible changes in the chemical and/or structural properties of Y , Zr , Pd , Pd-Au ally and polymers (PTFE, PMMA, FEP) before and after preparation, and how these properties changes as a function of an applied hydrogen pressure during hydrogenation, and oxygen during dehydrogenation.

Raman and Infrared (IR) signals of thin films of Y, Zr, Y-Zr alloys, Pd, Pd-Au alloys and polymers (PTFE, PMMA and FEP) were measured in the as-prepared state and in-situ in the presence of different concentrations of hydrogen. The samples were unloaded (dehydrogenated) in oxygen flow and the Raman and IR spectra acquired to check for possible changes in spectra. All measurement were done at room temperature using pure hydrogen and 5% H2/Ar mixture for hydrogenation, while dehydrogenation was done using a 20% O2/Ar gas mixture or just pumping air into the cell.

Major difficulties encountered was that the samples were not optimized for the particular Raman and and IR equipments at EMPA therefore the signals obtained was too weak (within the error limits of the equipments) to arrive at a reasonable conclusion. In particular the sample thickness was not high enough and the substrate (quartz) has a huge signal within the range of interest for our samples. We found out that the use of a different substrate than quartz for the thin film deposition could help to increase the signal to noise ratio.

Project 2021: Spectroscopic study of C-H vibrational modes in hydrogenated graphene

The objective was to integrate some neutron spectroscopy data sets of hydrogenated defective graphene (thermal exfoliated graphite oxide after thermal treatment in hydrogen flux) with IR spectroscopy and distinguish different C-H vibrational components, in the energy region corresponding to the C-H bending and stretching modes (100-150meV and 400 meV)

We collected several IR spectra on different hydrogenated graphene flakes and other similar carbon nanostructures (asprepared graphene and nanographite, the latter obtained by ball-milling of pure graphite) in order to evidence specific C-H groups in hydrogenated graphene. Measurements, as well as sample handling, were performed in controlled atmosphere, in order to prevent any possible oxygen/air contamination of the samples.

Data analysis, interpretation and peaks attribution in terms of C-H vibrations is still in progress and delicate in itself (very noisy data, low quantity of H, structural inhomogeneity of the sample, absorbant materials). They will be part of Chiara Cavallari's PhD thesis, as complementary characterization of the sample.

Project 2037: Catalytic combustion of hydrogen

In the cycle of production, storage and use of hydrogen, its catalytic combustion can play an important role to directly produce water and heat from a controlled combustion at temperatures typically below 500 oC without flame [1]. The catalytic reaction can be also used for safety reasons and should be efficient for low hydrogen concentrations and low temperatures. In addition we have recently designed and built at the laboratories in Seville a continuous reactor to produce hydrogen by sodium borohydride e hydrolysis at conditions to directly feed a fuell cell for portable applications. In this context the objective of our research for the long-term is to develop small devices coupling the already designed H2 generator to a heating system for portable applications. To achieve this final goal, medium and short term objectives are devoted to carry out fundamental studies as follows: i) Synthesis and microstructural characterization of catalysts and monoliths; ii) measurement of catalytic parameters for the combustion of lean H2 fuel-air mixtures.

A Pt-containing washcoat (supplied by INFRAGAS Srl) has been investigated as supported on a SiC-based foam (from LANIK Sro, 80 ppi) for catalytic combustion applications. A CuO/Al2O3 material was also investigated for comparison purposes. The work carried out at the LASY facilities included:

General access to main facilities in the laboratory during the complete period of stay.

- Specifically the use of a reactor for catalytic activity test of gas-solid reactions with temperature control and FTIR (Infrarred Spectroscopy) detection system for the quantification of water formation.
- Specifically the use of the chemistry laboratory for the synthesis of alternative catalysts by the co-precipitation methodology.

The main results already achieved concentrate on the study of the Pt containing washcoat catalyst supported on SiC foams. At the laboratory in Seville the complete microstructural characterization has been carried out. The ceramic SiC foam contains dense alumina particles as binder while the wash-coat is based on a high surface porous alumina that contains the dispersed catalytic phases (namely Pt and CeOx additive). This design has been shown to be well adapted for its application in a catalytic diffusion hydrogen burner at the EMPA laboratories. From catalytic tests measurements at the LASY facilities an activation energy of 34.6 kJ/mol has been measured for the coated foam material. For H2/air streams of 200ml/min at 24 oC (RT) a catalytic activity of 3.5 Nl H2 converted permin and per g of Pt in the coated foam material was found. The reactor is simulating real operation conditions without gas stream preheating or thermostatic stabilization of the reactor temperature. Catalytic activities higher than 18 Nl H2 converted per min and per g of Pt in the coated foam have been measured at temperatures below 50 oC. Finally the investigated CuO/Al2O3 material showed that Cu-based catalysts are not operating at room temperature. In this case temperatures over 200 oC were needed to measure appreciable conversions First communication of the results will be done next July at the Congress: ECNF2014 & Al-Nano Func Final Conference, Seville July 7th-11th, 2014.

The design of a reactor for the catalytic studies of the hydrogen combustion needs to consider the temperature control, as the reaction is highly exothermic; as well as the effect that water will condense at room temperature. To overcome these strategic points, and to adapt our reaction to the actual available facilities at LASY, the work was carried out using lean H2-air mixtures at conditions below the water saturation concentration in air at room temperature. The assistance and collaboration with the Technology Expert was very valuable.

Project 2050: Synthesis and characterization of novel metal amidoboranes

The aim of the proposed project is to contribute the development of a highly efficient chemical system for solid-state hydrogen storage, with a high potential to be transferred to a technology applicable for mobile on-board use.

Thus, the objectives of this project are as follows:

- synthesis of MAB adducts with ammonia borane of general composition MABn.mAB (where n is determined by charge of M);
- synthesis of bi-and trimetallic amidoboranes of the general composition M'M"M"'AB4 (where M' and M" are alkaline, while M"' is an alkaline earth metal, preferably Mg);
- characterisation of prepared compounds by means of XRD and vibrational spectroscopy;
- characterisation of their dehydrogenation by means of variable-temperature vibrational spectroscopy.

2. Description of installations provided

- <u>Laboratory for Solid Hydrogen Storage Materials (*Lasy*)</u>

  (a) Synthesis of Hydrogen storage materials

  (b) Characterisation of Hydrogen storage materials

  A variety of techniques can be offered to evaluate the key parameters of hydrogen storage materials, such as surface area determination by nitrogen adsorption measurements at 77 K: BELSORP-max (BEL, Japan), structural characterization by Raman spectroscopy as well as by in situ X-ray diffraction in various atmospheres (H_2, H_2O), and temperatures up to 800°C (Bruker D8). Gravimetric measurements are possible in inert atmosphere or under hydrogen of up to 200 bar in a modified RUBOTHERM magnetic suspension balance. The chemical composition of the desorbed gas are measured by thermal desorption mass spectroscopy (Balzers). The measurement of surface compositions is made possible by an X-ray photoelectron spectrometer (Specs). All sample preparation, handling and measurements are performed without contact to air.

Both tasks were used by users.

- Laboratory for the Development of Materials for Hydrogen production (*Lady*)

  (a) High Temperature Electrolysis by Solid Oxide Cells (SOEC)

  Structural investigations (PSD, grain growth and coarsening effects, pore size distribution and grain boundary aspects), determination of chemical changes, material interactions and diffusion processes after long operation time in the µm and nm scale. Phase changes as well as nucleation and growth of new phases can be analyzed by conventional XRD or into details by Synchrotron analysis.

  (b) Alkaline electrolysis (laboratory scale)

  Tests carried out using electrolysis test apparatus developed by the Electrochemistry group of the laboratory Hydrogen & Energy, provide the results on the diaphragm/membranes' gas separation properties and their impact on the cell voltage. Monitored hydrogen and oxygen gas purity, together with the cell voltage, are critical parameters for ranking the efficiency of the newly developed membranes.

  (c) Electrochemical characterization

  Electrochemical Impedance Spectroscopy (EIS) is suitable for gathering better understanding of the ion conductivity of diaphragms/membranes. For this purpose, a two compartment 4-electrode electrochemical cell controlled by Zahner IM6eX potenstiostat/galvanostat is used (Fig. 3). Moreover, electrochemical techniques, such as linear/cyclic voltametry, are undertaken in order to characterize the corrosion behavior of material.

  None of the infrastructures of Lady were used by the transnational access – due to overlap with other existing EU-projects; and due to problems with improvements of the installations.

3. Services offered

The specialized and experienced team of Hydrogen and Energy operates the above mentioned infrastructure. Within the framework of the project, access to the equipment will be provided for a determined period of time.

Generally, the potential user will provide the sample and the necessary information and the asked study will be performed by the hydrogen and energy staff. In the case of a highly experienced service demander, the user could perform part of the work, supported by the Hydrogen and Energy team.

Description of work lacking: please, describe the activity plan. IFE's description of work is a good example of the info expected here I have no idea, what you want from me . I can copy the description of work here – that's all we will do in this task