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## Synchrotron Radiation News

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t716100695>

## Energy Research at the ESRF

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Online publication date: 27 July 2010

**To cite this Article** Admans, Gary and Fitch, Andy(2010) 'Energy Research at the ESRF', Synchrotron Radiation News, 23: 4, 22 – 29

**To link to this Article:** DOI: 10.1080/08940886.2010.501492

**URL:** <http://dx.doi.org/10.1080/08940886.2010.501492>

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# Energy Research at the ESRF

Edited by GARY ADMANS AND ANDY FITCH

European Synchrotron Radiation Facility, Grenoble, France

The modern world consumes ever increasing amounts of energy to sustain human numbers and contemporary lifestyles. Research and development of energy supplies and their exploitation are consequently topics of major economic, political and scientific importance. Moreover it is clear that current reserves of energy, predominantly fossil fuels, are limited, and there is widespread apprehension about the climatic effects of returning large amounts of carbon dioxide and other green-house gases to the atmosphere. Thus there is a degree of urgency to such studies. Energy systems are often of a complex nature. Development of new energy sources and improving the efficiency and exploitation of existing systems require detailed understanding of both structure and behaviour at the fundamental microscopic level, an area where the powerful X-ray beams of a synchrotron radiation source can play a major role. Thus energy research has become a theme of great scientific and practical importance at institutes such as the ESRF, which serves the scientific priorities of the research communities of its member countries.

In this report we present a few examples of recent research, a subset of the many other studies carried out on ESRF beamlines by users of the facility. Topics illustrated include nuclear energy, where X-rays help make current nuclear technology safer and cleaner, while preparing the groundwork for future generations of fusion reactors. Making the most of existing resources is another challenge. How do we make good use of the dwindling supplies of oil, and how best to convert sustainable biomass into fuel for transportation? Solar cell technology improves continuously. X-ray studies are used to characterize silicon wafers, to help optimize their manufacture and make them more reliable, and for fundamental studies of new solar-energy materials. Technologies based on hydrogen also show promise, but there are constraints to be overcome in storing and using this fuel. X-ray studies provide insight into the conditions at the center of a working fuel cell and help develop new components.

Imaging, diffraction, and spectroscopy allow detailed investigation of the structure of materials and their behavior while processed *in situ*. The variety of sample environments at beamlines is one key to the diversity of research. Specific reaction chambers mimicking industrial plants make possible *in operando* studies allowing scientific insight at the heart of a reaction process. These are some of the features of modern synchrotron research that enhance today's energy research and technology for a cleaner and more productive future.

## Nuclear energy

Nuclear fission supplies around 13–14% of the world's electrical power, and in the future there are hopes that nuclear fusion could supply

a significant amount of energy, without the problems associated with reprocessing or storage of high-level nuclear waste. There are many materials challenges in the development of nuclear fusion to withstand the extreme conditions that will exist inside an operating reactor.

Plasma-sprayed tungsten is a porous coating with promising thermo-mechanical properties applied to surfaces directly exposed to the fusion plasma, where it has to withstand high heat flux and thermo-mechanical stress. Accurate micromechanical modelling is required to understand the complex mechanisms governing damage and failure of the coating. As input for these calculations, it is necessary to know the statistical properties of the pore microstructure at the boundary between the two metals. While two-dimensional information can be obtained from electron microscopy, the only way of obtaining three-dimensional volume data of actual porous networks is via X-ray microtomography. The challenge of such a study is the high spatial resolution required (on the order of 1  $\mu\text{m}$ ), combined with the high density and high atomic number of tungsten, which make it necessary to use relatively high photon energies while keeping the sample size small. At beamline ID19, 52-keV X-rays were used in a tomography setup with a pixel size of 1.4  $\mu\text{m}$ . The samples had to be machined to a cylindrical shape, with a diameter of approximately 0.5 mm. Figure 1 shows three-dimensional renderings of a region of interest in one of the samples investigated [1].

Another study towards the development of fusion reactors was carried out at ESRF beamlines ID15 and ID19. Researchers used microtomography to look at the internal structure of model pebbles for the helium cooled pebble bed (HCPB) blanket design reactor. With this technique the number of contacts between aluminium pebbles (used for

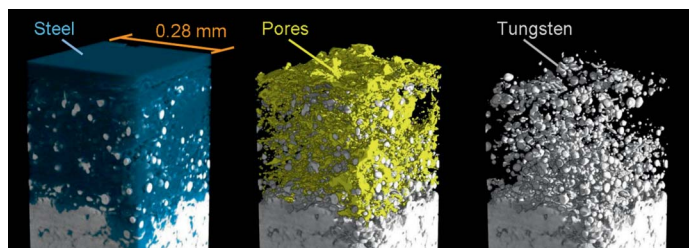
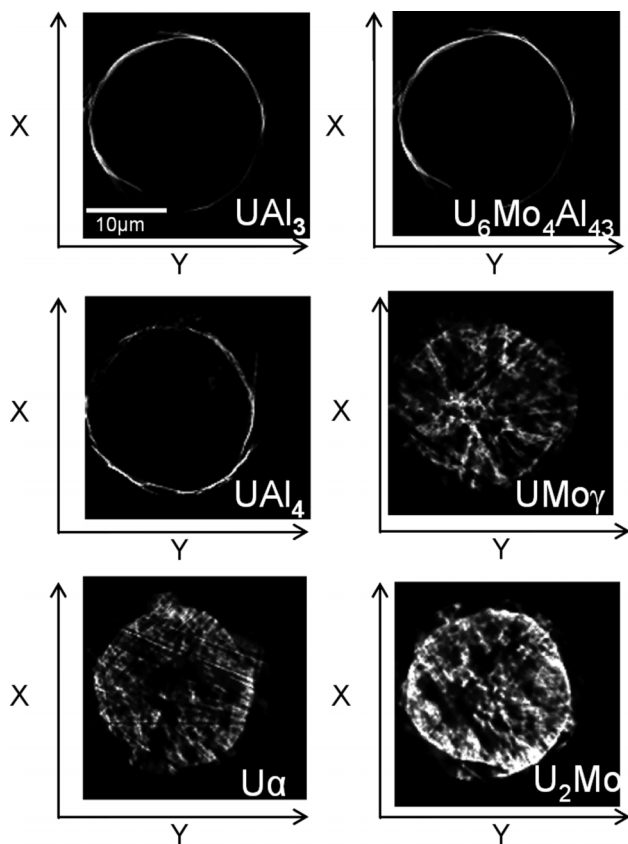


Figure 1: Three-dimensional renderings of a tungsten-stainless steel interface (tomography using 52-keV X-rays, voxel size 1.4  $\mu\text{m}$ ). The tungsten layer is in light grey, the steel substrate in blue and the extensive pore network in the mixing zone highlighted in yellow (image courtesy of T. Weitkamp [1]).

better measurement accuracy instead of beryllium pebbles) and their angular distribution were obtained as well as the corresponding contact surfaces. The pebbles were also subjected to the effect of a simulated plasma excursion at high pressure. This permitted evaluation of tritium bubble sizes (produced during reactor functioning) and densities in single pebbles, the pore channel network topology, the 3D reconstruction of the fraction of interconnected bubble porosity, and the open-to-closed porosity ratio [2,3].

X-ray studies are also helping to improve fission reactors. New generations of nuclear fuels for fission are developed in dedicated material testing reactors. In-reactor experiments are carried out to test and characterize the new materials. Highly enriched fuel materials are typically used in this kind of reactor. However, for non-proliferation purposes, a world-wide program encourages the development of low-enriched nuclear fuels (below 20%), with the most promising being high-density U-Mo/Al fuels.



**Figure 2:** UMo particle extracted from an annealed fuel plate (*xy* plane, 25  $\mu\text{m}$  in diameter). XRD-CT using nano X-ray beams shows a 1- $\mu\text{m}$ -thick interaction layer around the UMo particle consisting mainly of  $\text{UAl}_4$  or  $\text{UAl}_3$  and  $\text{U}_6\text{Mo}_4\text{Al}_{43}$ . Within the particle core, partial decomposition of the  $\gamma$ -UMo phase leads to the appearance of two new phases:  $\alpha$ -U and  $\text{U}_2\text{Mo}$  (image courtesy of H. Palancher [5]).

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UMo fuel elements usually consist of spherical particles (with a diameter ranging from 30 to 120  $\mu\text{m}$ ) dispersed in an Al matrix. However, their in-pile behavior is currently not satisfactory because of the growth of a large interaction layer at UMo/Al interfaces while under irradiation. A breakthrough in this fuel development has been accomplished recently by adding a small concentration of Si to the Al, leading to a significant reduction in the size of the interaction layer.

To simulate the influence of in-pile irradiation or manufacturing conditions (hot-rolling), thermal annealing methods are usually performed and then the crystallographic composition of the resulting interaction layer studied. Because the interaction layer can have a thickness below 2  $\mu\text{m}$ , the most suitable non-destructive technique permitting its study is X-ray diffraction computed tomography with nano X-ray beams ( $0.2 \times 0.2 \mu\text{m}^2$ ) [4,5]. This 3D method is available at ESRF beamline ID22NI. Figure 2 presents an example of characterization of the interaction layer for a UMo particle.

### Fossil fuels

Fossil fuels continue to supply the majority of energy for the industrialized world. The negative impact on our planet of using these fuels is well documented. Research by users of the ESRF is helping to make

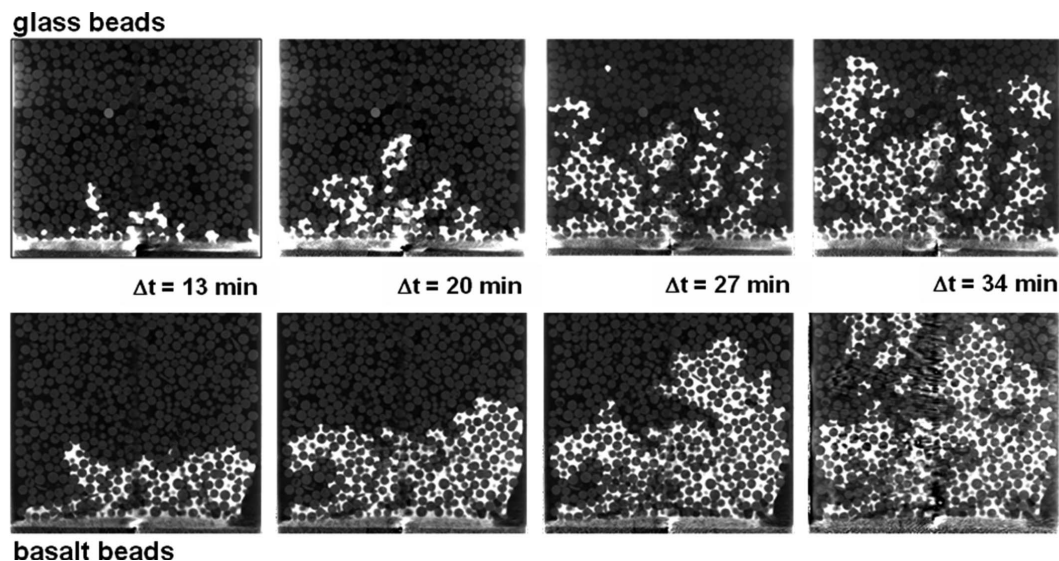


Figure 3: Comparison of water flow into oil supported on glass and basalt beads. Each image is a cross-section through a three-dimensional tomography scan. Water (containing  $ZnI_2$ ) was pushed into oil (dodecane)-saturated bead packs. The glass beads (top row) are preferentially wetted by water; the basalt beads (bottom row) are preferentially wetted by oil. The water front advancing into the basalt bead pack is more compact. Sphere diameter is 300–355  $\mu m$ , imaging time was 3 s per scan, and the average front velocity was about 50  $\mu m/min$  (image courtesy of R. Seemann [6]).

better use of existing fossil fuel supplies, as well as aiding in the development of processes permitting renewable sources of energy and their conversion into biofuels.

Crude oil is recovered from porous rock, which is (partially) filled with oil. Primary oil recovery permits 5 to 20% of the stored oil to be recovered by pumping the oil from the wells. When the natural reservoir pressure dwindles, the reservoir has to be artificially pressurized. This is typically done during the secondary oil recovery by drilling additional wells and injecting water into the reservoirs. By pushing water into the reservoir, the extracted oil is replaced by the injected water and ideally a water/oil front is pushed towards the production well. However, the higher viscosity of the oil often results in the water finding preferential flow paths through the reservoir and the water can under-ride the oil. Even in an ideal setting the displacement of the oil by water is quite complex and not piston-like. The multiphase flow of water and oil in the porous rock varies with the wettability of the rock, its porosity, the viscosity and chemical composition of the crude oil, and the injection speed of the water. During the secondary oil recovery up to 50% of the initially stored oil will be recovered.

To better understand the complex multiphase flow of two immiscible liquids on the scale of single pores, an investigation was carried out at ESRF beamline ID15 using ultrafast time-resolved X-ray tomography (Figure 3) [6]. This study will eventually help in increasing the recovery factor of oil from reservoirs. Moreover, the experimental techniques and the studied flow behavior are similar to evaporation or absorption of water from or into soil, impregnation effects, soil contamination with

organic spills and its decontamination, and  $CO_2$  sequestration, thus allowing insights into these important phenomena.

Fischer-Tropsch synthesis is a key step in the production of fuel or chemicals from carbon sources alternative to crude oil, such as gas, coal or biomass. Commercial plants employing this process typically convert natural gas to high-quality diesel. The reaction involves the catalytic conversion of syngas, a mixture of carbon monoxide and hydrogen, into long-chain hydrocarbons. The catalyst is usually iron or cobalt and reaction conditions involve high temperatures and pressures. Although well proven and used on a very large scale, the industrial process does have complications such as gradual deactivation of catalysts. Research at ESRF beamlines has provided insight into this reaction *in situ*, using reaction cells designed to mimic industrial reactors. At ESRF CRG beamline BM01 (SNBL), the structure–activity relations for alumina-supported cobalt catalysts were studied under realistic conditions using time-resolved X-ray diffraction and catalytic measurements [7]. Two processes for deactivation of the catalyst were witnessed, sintering of the cobalt and carbidization. Further information was gained in a study whereby XANES was used to confirm the oxidation state of the cobalt species under similar reaction conditions [8]. On ESRF CRG beamline BM26 (DUBBLE), another team of researchers has been investigating iron-based catalysts, which are promising for biomass conversion [9]. They used *in situ* XAFS and WAXS to look at the iron species present in the reaction for supported and unsupported catalysts. They also found suitable pre-treatment conditions for the catalyst that gave higher reactivity in the catalytic synthesis. These

three academic studies have increased our knowledge about a reaction that is becoming more important as reserves of fossil fuels diminish. Each represents a small step towards the development of cleaner industrial processes, and looking further ahead may lead to a means of producing a greater proportion of tomorrow's fuels from renewable sources.

**Electrical energy**

Synchrotron X-rays permit imaging at the micro- or nanometer scale with trace element sensitivity and also real time experiments. In the next two examples, first sub-micrometer-scale fluorescence maps are used to identify the elements present in silicon solar cell at grain boundaries and dislocations. In the second example, it is the high penetrating power of hard X-ray beams that makes it possible to monitor conditions within a working fuel cell.

Diode breakdown in multicrystalline silicon solar cells from novel feedstock materials is a problem that has forced manufacturers to adapt the module design to decrease breakdown behavior of the resulting solar cells. Three different breakdown mechanisms have been identified [10]. One is related to recombination centers in the Si crystal that are caused by metal decoration of crystallographic dislocations and grain boundaries. X-ray  $\mu$ -fluorescence (XRF) mappings were made

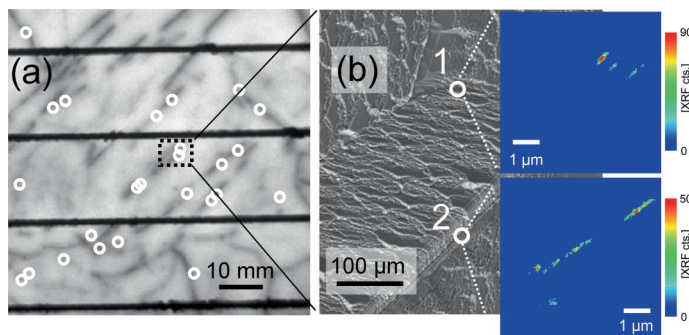


Figure 4: a) Detailed view of an electroluminescence image of a standard industrial multicrystalline silicon solar cell. The horizontal lines correspond to the front side contacts. The rather hazy features in between are caused by recombination of active defects such as grain boundaries. Pre-breakdown light emission is observed at all sites marked by white circles. b) SEM image of the section marked by the dotted rectangle in (a). Inset images:  $\mu$ -XRF maps of the Fe- $K\alpha$  line intensity around sites 1 and 2. At both pre-breakdown sites, Fe precipitates are clustered along the grain boundaries (image courtesy of G. Martinez-Criado [11]).

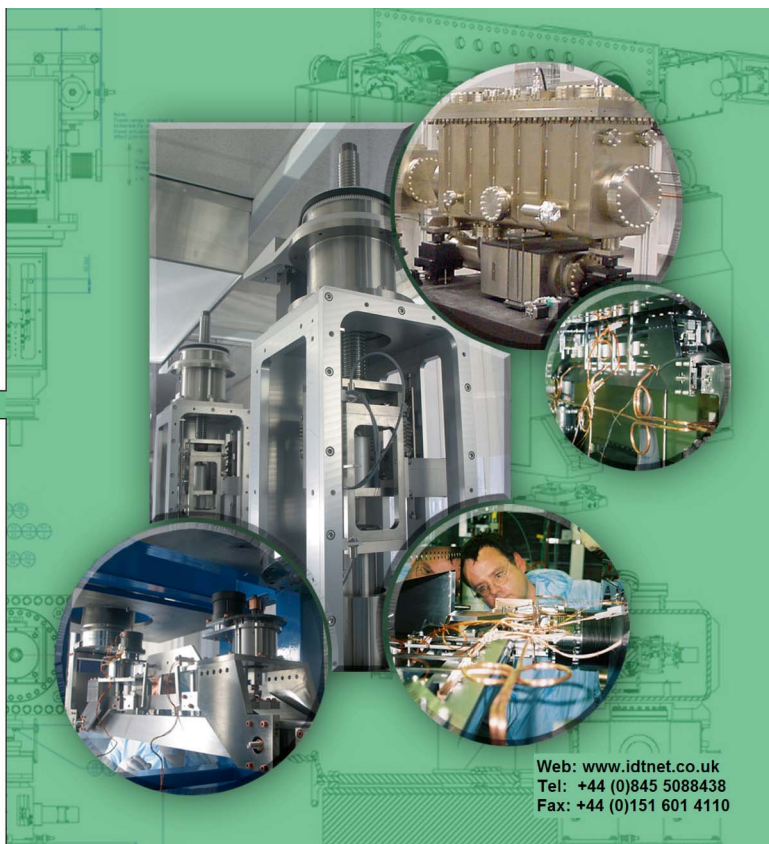
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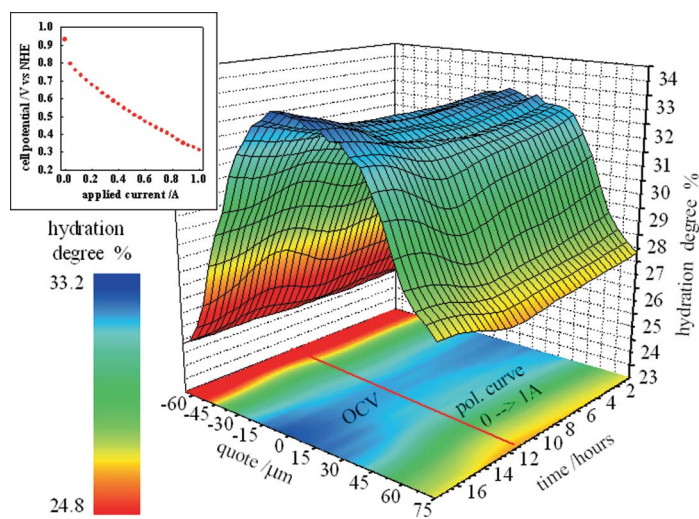


Figure 5: Space-time distribution of water inside the proton-exchange membrane, obtained by cyclically scanning the membrane from the anode to the cathode during a polarization curve (current ramp up to 1 A). After the end of the ramp, the circuit was opened to observe the relaxation to the hydration equilibrium value (image courtesy of B. Paci [13]).

using the nanoprobe at ESRF beamline ID22NI for a silicon solar cell showing pre-breakdown spots. The results are shown in Figure 4. Iron precipitate colonies were only detected at the locations of the breakdown spots along a grain boundary. Thus pre-breakdown related to recombination of active defects is solely caused by metal precipitates [11]. This finding suggests that this breakdown type is closely linked to precipitate-related properties, such as the formation of a Schottky contact-like barrier between metal cluster and silicon.

Research into promising materials for high-efficiency thin-film solar cells is also an active area. At beamline ID15, powder diffraction experiments on polycrystalline  $\text{Cu}_2\text{ZnSnS}_4$  (a novel semiconductor) have revealed a high-temperature phase change [12]. To carry out such studies, a furnace was used, permitting temperatures of up to  $1100^\circ\text{C}$ . The findings from this research are important for the functioning and manufacture of thin film devices.

Fuel cells are electrochemical devices that convert chemical energy into electrical energy. The most promising designs are probably those based on proton-exchange membranes (PEMs), whose possible applications range from power generation plants to locomotion. Nevertheless, several technical issues must be solved before such fuel cell systems can become a commercial reality.

PEMs need to be suitably hydrated since their proton-conducting properties are related to their degree of hydration. However, an excess of water can be detrimental, and eventually lead to flooding and cell failure. The mechanisms involved in the PEM's water uptake and removal have been studied *in situ* by means of high energy X-ray diffraction [13], e.g. with a beam of about 90 keV at ESRF beamline

ID15. The use of such hard radiation has permitted many experimental difficulties to be overcome: the beam has to cross a 5-cm-thick layer of X-ray absorbing material in the fuel cell and both the membrane and the water contained within are non-crystalline.

Diffraction patterns were obtained in short intervals, thus enabling a fine temporal sampling, even when an extremely thin beam was used (below  $10\ \mu\text{m}$ ). As an example, the 3D plot in Figure 5 shows the time-dependent distribution of water across the membrane. The surface thus obtained gives a direct insight into the degree of hydration of each part of the membrane, allowing observation of the effect of any change of the working parameters, both spontaneous or induced, as well as the characteristic response time to a change. These experiments permitted PEMs of fuel cells to be studied while working under various operating conditions such as different electrical parameters, temperature, gas flux, and pressure exerted on the PEM by the other cell components.

X-ray studies are also helping in the development of new fuel cell components. Nafion membranes have the disadvantage of being limited to temperatures of  $90^\circ\text{C}$ . At ESRF CRG beamline BM26 (DUBBLE), researchers have investigated an alternative material with high-proton conductivity at elevated temperatures [14]. Highly sulfonated poly(p-phenylene sulfone)s were found to have seven times higher proton conductivity at  $135^\circ\text{C}$  and 30% relative humidity than Nafion under similar conditions.

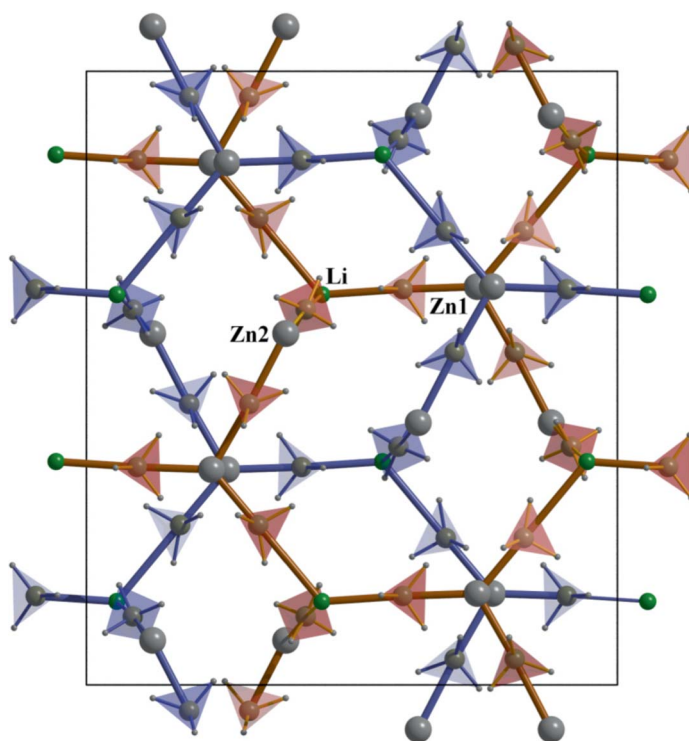


Figure 6: Crystal structure of  $\text{LiZn}_2(\text{BH}_4)_5$ . The doubly interpenetrating 3D framework is shown in blue and red (image courtesy of Y. Filinchuk [17]).

### Hydrogen and gas storage

Hydrogen is a clean fuel alternative for transportation via combustion or generation of electrical energy in fuel cells. Under investigation at synchrotron sources are materials capable of storing this gas so that it can be used away from the site of production in a safe manner – not an easy task given the volatile nature of hydrogen gas.

Light-weight hydrides are seen as future hydrogen storage materials: they have high hydrogen capacity and some desorb hydrogen reversibly. Diffraction studies of structures and transformations lead to an understanding of the solid state chemistry and properties of such hydrides.

Studies based on pressure and temperature changes can reveal new polymorphs of a hydride and their structural evolution. X-ray powder diffraction, high-pressure techniques utilizing diamond anvil cells, DFT calculations and a symmetry-based analysis were used with ESRF CRG beamline BM01 (SNBL) to study the  $\text{NH}_3\text{BH}_3$  and  $\text{M}(\text{BH}_4)_n$  systems. Directional ionic-covalent  $\text{M} \dots \text{BH}_4$  and dihydrogen  $\text{N-H} \dots \text{H-B}$  bonding were revealed [15,16]. Understanding the structure makes it possible to design new hydrides with better thermodynamic stability. The temperature of hydrogen desorption could be reduced to a more favorable level (80–100°C) in borohydrides containing a combination of alkali and transition metals such as the Zn-based borohydrides, e.g.  $\text{MZn}_2(\text{BH}_4)_5$  (M = Li, Na) [17] (see Figure 6). Other chemical derivatives were

prepared by solid-state synthesis and their stability and decomposition reaction pathways studied by *in situ* powder diffraction. The most recent novel compositions included bimetallic  $\text{MSc}(\text{BH}_4)_4$  (M = Li, Na, K) [18],  $\text{Li}_4\text{Al}_3(\text{BH}_4)_{13}$ , transition metal borohydrides  $\text{Y}(\text{BH}_4)_3$ ,  $\text{Mn}(\text{BH}_4)_2$ , and mixed-anion  $\text{Li}(\text{BH}_4\text{Cl})$  and  $\text{KZn}(\text{BH}_4)\text{Cl}_2$ .

The optimization of materials under realistic conditions was also investigated using *in situ* powder diffraction. The kinetics and thermodynamics of parallel reactions, hydrogen cycle stability of various Mg-based systems, including Mg-La [19], Mg-Fe-Co [20] and Mg-V, have been studied. The reversibility of borohydride-based composites with high hydrogen content has also been demonstrated.

Another framework for storing gas molecules are the clathrate hydrates. These are ice-like inclusion compounds where guest molecules are embedded in a hydrogen-bonded water network. Hydrates have reached public interest because they are promising candidates for gas storage, e.g. for  $\text{CO}_2$  sequestration or hydrogen storage for fuel cells. However, their formation on molecular length scales is still under investigation. In general, two different models exist describing the formation process, in a liquid mixture or at the water surface via hydrate precursors, or via a stochastic arrangement of water and guest molecules without precursors, respectively. Thus, both models predict a different local structure of the water molecules, which is accessible by



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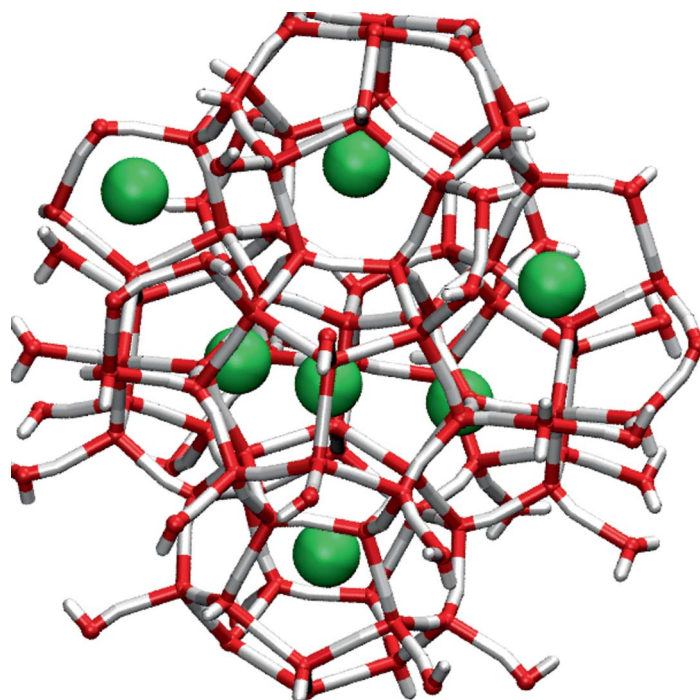


Figure 7: Schematic drawing of a clathrate hydrate structure. The green spheres represent the guest molecules within the host network (image courtesy F. Lehmkuhler [22]).

various X-ray scattering techniques. Using ESRF beamline ID15, the formation process of CO<sub>2</sub> hydrates was studied at the water-CO<sub>2</sub> interface by X-ray reflectivity and diffraction measurements. Tetrahydrofuran (THF) hydrates from a water-THF mixture were also studied by inelastic X-ray scattering. At the water-CO<sub>2</sub> interface only adsorption of CO<sub>2</sub> onto the water surface was observed while hydrate precursors or crystallites cannot be detected. In contrast, the water-CO<sub>2</sub> liquid-liquid interface exhibits the instantaneous formation of small and mobile hydrate crystallites as observed by X-ray diffraction. The studies on THF hydrate formation exhibited no significant deviations from the expected temperature effect on the inter- and intramolecular bonds. Again, this result suggests no signature of hydrate precursors, which is supported by density functional theory calculations. Hence, similar to CO<sub>2</sub> hydrate, a stochastic formation model is favored for THF hydrate. This knowledge of the hydrate formation process may have a large impact on the synthesis of hydrates for gas storage applications and also to inhibit the formation of hydrates, e.g. to prevent plugging of gas and oil pipelines by clathrate hydrates.

### Outlook

The ESRF is evolving thanks to an Upgrade Programme [23]; the first part aims to deliver eight new beamlines and the refurbishment of many existing beamlines. The Upgrade focuses on five core areas of

science: nanoscience and nanotechnology; pump-probe experiments and time-resolved diffraction; science at extreme conditions; structural and functional biology and soft matter; X-ray imaging. Many of these areas will further the possibilities for energy research. Imaging on the nanoscale promises to add even greater precision for tomographic studies. Smaller, more intense, high-energy beams will offer greater penetration depths, enhancing engineering studies. Improved time resolution will enhance *in situ* studies of chemical reactions, giving greater detail about the processes occurring at the atomic and molecular level. These, as well as many other new developments, will make it possible for the pioneers of energy research, our users, to make even greater discoveries through their investigation and innovation. The ways in which we will all use energy in the future depends critically on their successes. We wish them the best of luck and we thank them for their efforts.

### Acknowledgements

The editors would like to emphasize that this paper is based on the work of a large community of ESRF staff and users as seen from the long list of authors cited in the references. We wish to acknowledge the contribution of all of these colleagues to the scientific and technical advances presented in this paper.

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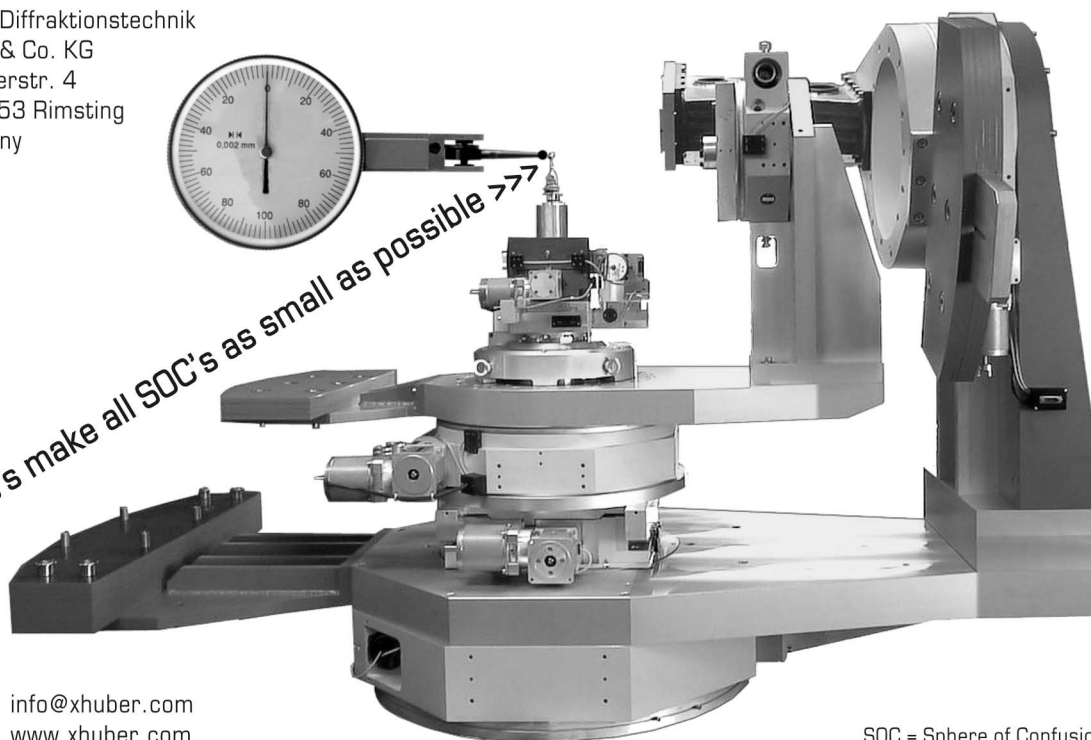


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