

REPORT

ESS Science Symposia "Time-resolved, in-situ chemistry using neutrons" Chalmers University of Technology, Gothenburg, Sweden, April 26-27, 2012

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Report on the ESS Science Symposium “Time-resolved, in-situ chemistry using neutrons”, Chalmers University of Technology, Gothenburg, Sweden, April 26-27, 2012

Summary and conclusions

- One should work for the construction of a “Sample cell development complex” at the ESS site side by side with the construction of ESS and its instrument suite
- The “complex” will be the natural meeting place for ESS facility people and University staff and as such promote interaction between ESS and Universities
- The “complex” will favour joint efforts providing the best of knowledge from science users and facility know-how (including access to good engineers, workshops, safety issues)
- One should encourage interaction and exchange of know-how between neutron facilities (e. g. ESS, ILL, ISIS, HMI)
- In order to strengthen the neutron scattering community, and focus more on time-resolved in-situ chemistry, relevant summer schools should be arranged
- ESS, Research Councils, and Universities need to start discussions in order to find possible ways to fund the concept of a “Sample cell development complex”
- An “advisory panel” should be formed with the aim to discuss and develop the ideas expressed during the Symposium, both with respect to maximizing interaction between ESS, other large scale facilities and Universities, as well as to identify funding routes

Background and aims of the Symposium

The solid state chemistry community was brought together in order to discuss scientific issues to be solved by access to a state-of-the-art neutron facility with an emphasis on the need for complex sample environment cells (SECs).

The aims were to identify the science drivers for in-situ studies, requirements and current state of the user community and, if possible, make a 'wish-list' of neutron friendly sample environments and get the developers together in order to identify the best funding routes to progress this work side by side with the construction of ESS and its instrument suite. On the agenda was also to discuss the role of existing large scale facilities (like ILL, ISIS, HMI). In this context the aim was to consider different possibilities to organise the science-driven development work, and how the “sample cell development infrastructure” should be organised, including both facilities and universities (see Appendix 1).

The invited speakers covered a wide range of topics (see Appendix 2) and expertise and included Thomas Hansen (ILL), Michael Simonson (Oak Ridge National Laboratory), Bill David (ISIS), Steve Hull (ISIS), Dirk Wallacher (Helmholz-Zentrum Berlin), Paul Henry (ESS), Pascale Deen (ESS), Mogens Christensen (Aarhus), Duncan Gregory (Glasgow), Dermot O`Hare (Oxford), Anthony Powell (Heriott-Watt), Johan Gustafson (Lund). In total approximately 30 persons took part in the workshop and the discussion sessions (see Appendix 3).

Topics discussed

The focus of the symposium was time-resolved in-situ chemistry using neutrons and to obtain a picture of the present status of the state-of-the-art when it comes to complex sample environments. We also considered how to organize and spread information amongst users, or potential users and facility staff in order to optimize interaction between universities and facilities. Of importance will be to find channels for funding of joint efforts like this.

Historically facilities and neutron instrumentation at facilities have been designed and developed independently of the development of sample environment cells. Our message is that one should coordinate beamline design, instrument and sample environment development as much as possible. There must already at an early stage be resources available for the sample environment design in order to make it possible to optimize instrument and sample environment as much as possible with respect to each other.

The in-situ studies performed must always be science driven, the science-drivers identified, and technical problems solved. It is therefore important to exploit key-competences at both facilities and universities/research institutes to maximize the outcome. The already existing large scale neutron facilities (*e.g.* ISIS, ILL, HMI) do normally have well developed infrastructure when it comes to access to workshops, technical support and user support. All of these factors are of importance when it comes to a simultaneous or more efficient development of neutron instrumentation and sample environment cells and an improved interaction with external users (normally with university affiliation but also industrial contacts). When it comes to sample cell design it feels natural to focus the development activities mainly to the facilities but with a strong link to universities, or key-persons/groups at universities. In order to promote information flow and exchange between facility and university staff (normally users) one should create a "Sample cell development complex" (see next paragraph) connected to ESS. A joint effort provides the best of knowledge from science users and facility know-how (including access to good engineers, workshops, safety issues), *e.g.* to develop sample chambers with a focus on in-situ chemistry in the neutron beam. Similar arrangements may be of interest to develop at other neutron facilities as well. A "complex" of this kind could also be a way to organize and strengthen the in-situ or solid state chemistry community.

A "Sample cell development complex"

From our perspective, when it comes to ESS in Lund, a "Sample cell development complex" or a "Sample cell development building" should be an ideal way to promote interaction between universities and the facility already from the very beginning. Such a "complex" will efficiently link scientific activities with technical development and testing. The main focus should be on development of sample cell environments of high scientific interest. A number of key-persons employed by ESS should be connected to the "complex" and act as contact persons towards universities and university groups. The key-persons should include instrument scientists, engineers and technical staff and will naturally have strong links to for example workshops and other support units at ESS. Their responsibility should for example be to discuss, judge and coordinate suggested sample cell development projects together with external groups, *i.e.* conclude if a project is of high scientific interest, technically feasible, etcetera. To run development projects within one "building" or "complex" have many advantages. One can in a more efficient way use resources, people will learn from each other, and there will be synergistic effects between projects. It will also be easier to survey running projects and avoid overlapping work and to inform about on-going activities to the neutron scattering community and potential users. Once or twice a year meetings should be arranged to inform about new achievements.

The "complex" should in principle work as an outstation for universities making it possible for senior scientists, post docs and PhD students to spend time at the facility (possibly funded by research grants, faculty money, or special programs). Selected persons from the university side should spend substantial time at the facility (based on experience around 70%) and be responsible for development of specific sample environments in close collaboration with instrument scientists, engineers and technical staff. There must be a strong scientific element connected to the development work, particularly if PhD students and post docs are involved. The "complex" should, in addition to resources for sample cell design, provide with basic equipment (to be decided by ESS and user representatives) making it possible for PhD's and post docs also to focus on science projects (in collaboration with ESS staff and their home university). There will also be a need for technical support and maintenance of key-equipment and laboratories.

The main activity within the "complex" will obviously be the design of sample chambers. Although it is a bit early to suggest specific designs (beamlines must be defined in more detail first) an interest for more and more complex sample environments is expected. One can for example consider collecting neutron diffraction data as a function of temperature and controlled gas mixing, and at the same time monitor oxygen partial pressure, and measure conductivity and Raman scattering. The goal should always be to design sample environment cells possible to use at different instruments without compromising the quality of data collected.

The "Sample cell development complex" should provide with the infrastructure needed, and act as a meeting point for scientific exchange. An important task will be to inform the science community about activities, and thus raise the interest for neutron scattering to a broader audience, and promote an increased scientific and technical exchange between ESS and the user community in general. The construction and operation of a complex of this kind demand large investments. Our belief is that ESS, different research councils and universities need to start discussions in order to find possible ways to fund an infrastructure of the kind briefly described.

Sample cell development – what can we standardize

An ideal situation would be if one can use sample cells not only at a specific instrument at a specific neutron facility but at different instruments, or even different facilities, or at a "home" university. Many times this will not be technically feasible but one should consider for example to use standard electric connections or connections for data transfer. Development and use of instrument control- and data handling software should be standardized whenever possible. The starting point should be to standardize as much as possible within ESS.

Development and use of sample cells, responsibilities

Standard sample cells will normally be provided by the facility. It can be furnaces or cryogenic equipment for temperature studies. Maintenance and support for users is relatively straightforward and part of the facilities obligations.

When it comes to non-standard equipment, especially during the development phase, there is a demand for extra resources. The equipment should mainly be developed at the ESS facility but in close collaboration with external groups connected through the "Sample cell development complex". For sample cells of common interest users could apply for facility funding in order to take part in joint sample cell development activities. Generally our feeling is that the facility should be responsible for, and take care of the sample cells developed. One should, however, from case to case write

agreements in order to avoid misunderstandings when it comes to ownership, and responsibilities for the equipment.

It is of importance to encourage people to be involved in sample cell development work. People involved need to get credit for their efforts as it is quite a time consuming job to design, construct and commission new sample cells. Credit can be given in different ways, for example by being author of first publications using the new equipment and by being cited.

Education of people - Summer schools

The importance of a growing neutron scattering community cannot be overestimated. For this purpose it is important to find the appropriate structures for education of people, both new students and more experienced neutron users. Summer schools covering both theoretical and practical aspects of neutron scattering and elements covering sample environment and in-situ studies in the neutron beam should be arranged (to our knowledge not yet arranged). Such a combination should be very attractive for many users. Part of the education can be held at Universities but there is also a need to get hands-on experience at a neutron facility.

Sten Eriksson

Stefan Norberg

Steve Hull

Paul Henry

Time-resolved, in-situ Chemistry using neutrons

26-27 April 2012, Gothenburg, Sweden

Programme

The Symposium will start Thursday 26 April at 13:15 and end in the early afternoon Friday 27th. A short introduction and update of the status of the European Spallation Source will be given by Paul Henry (ESS, Lund). There will be scheduled time for oral sessions and for discussion in smaller groups. More detailed information will be provided before the meeting and upon registration (see below).

Aim of the symposium and anticipated outcome

The workshop will bring the solid state chemistry community together in order to discuss scientific issues to be solved by access to a state-of-the-art neutron facility with an emphasis on the need for complex sample environment cells (SECs).

The idea is to promote an exchange between facility staff and neutron users, or potential users, with a scientific interest in fuel cells and batteries, mapping of electrochemical reactions, hydrothermal reactions, study solid-gas phase reactions, reactions under controlled atmosphere or humidity, just to mention a few examples.

The aims are to identify the science drivers for in-situ studies, requirements and current state of the user community and a 'wish-list' of neutron friendly sample environments and get the developers together so we can identify the best funding routes to progress this work side by side with the construction of ESS and its instrument suite. One should also in this context discuss different possibilities to organise the science-driven development work, and how the scientific infrastructure should be organised.

The expected outcome will be to:

- Identify the most important needs for facility staff as well as the scientific community
- Suggest guidelines for improved interaction between facility staff and the scientific community by identifying suitable structures promoting an exchange
- Suggest development of a range of SECs based on the demand from the scientific community (with technical limitations in mind)
- Suggest how to promote and optimise interaction between different neutron facilities and groups at different universities as well as industrial partners
- Submit a workshop report with a wish list of sample environments, suggestions for funding routes and how to organise and optimise an infrastructure for the science-driven development work

The invited speakers cover a wide range of expertise and include Thomas Hansen (ILL), Michael Simonson (Oak Ridge National Laboratory), Bill David (ISIS), Steve Hull (ISIS), Dirk Wallacher (Berlin), Duncan Gregory (Glasgow), Dermot O`Hare (Oxford), Anthony Powell (Heriott-Watt)

Venue and registration

The Symposium will be arranged in Gothenburg at Chalmers University of Technology, Building MC2, Lecture room Kollektorn.

We welcome you to send in contributions, we still have slots for a few additional oral presentations and posters will be shown during the Symposium. Additional information will be provided upon registration.

For registration and support with accommodation please contact Charlotte Bouveng (Bouveng@chalmers.se).

Organisers: S. Eriksson (Chalmers, Gothenburg); S. Norberg (Chalmers, Gothenburg); P. Henry (ESS, Lund)

**ESS Science Symposia: Time-resolved, in-situ chemistry using neutrons,
Gothenburg, Sweden, April 26-27, 2012**

Thursday, April 26

Time: 13:15 – 19:00

Lecture Hall Kollektorn, MC2, Chalmers

13:15 – 13:20	<i>Introduction and welcome</i> Sten Eriksson, CTH
13:20 – 13:50	<i>European Spallation Source, Lund</i> Paul Henry, ESS AB
13:50 – 14:20	<i>In-situ Studies at ISIS: SOFC Materials</i> Steve Hull, ISIS
14:20 – 14:50	<i>Spectroscopy and Time-resolved Diffraction for Chemical Systems</i> Mike Simonson, Oak Ridge National Laboratory
14:50 – 15:20	COFFEE
15:20 – 15:50	<i>Parametric and time-resolved constant wavelength neutron powder diffraction at D20</i> Thomas Hansen, ILL
15:50 – 16:20	<i>Studies of hydrothermal reactions at synchrotron and neutron sources</i> Dermot O'Hare, University of Oxford
16:20 – 17:30	Discussion I) in groups, specified topics, including summary afterwards
17:30 – 18:00	COFFEE
18:00 – 18:30	<i>In situ catalysis on the atomic level</i> Johan Gustafson, Lund University
18:30 – 19:00	<i>Ultra-rapid solid state materials synthesis; probing microwave reactions ex- and in-situ</i> Duncan Gregory, University of Glasgow
19:30	DINNER

Discussion I)

The focus will be on infrastructure, if, and in such a case how “sample cell device” projects should be coordinated between facilities, universities. Needs at facilities, and universities will be discussed. Responsibilities, if it should change with time, *i.e.* during design/construction phase and “harvesting” phase? How to fund activities of this kind is of course an important part, is joint applications facilities/universities a way to go? How can one make people interested, attract more potential users? Good ways to spread information?

Moderator: Thomas Hansen, ILL

Friday, April 27

Time: 09:00 – 13:15 Lecture Hall Kollektorn, MC2, Chalmers

09:00 – 09:30	<i>An overview of direct geometry spectroscopy at the ESS</i> Pascale Deen, ESS AB
09:30 – 10:00	<i>Combined gravimetric analysis and neutron powder diffraction</i> Bill David, ISIS
10:00 – 10:30	<i>Diffraction-Resistance Measurements: Simultaneous Investigation of Structure, Magnetism and Transport Properties</i> Anthony Powell, Heriot-Watt University
10:30 – 11:00	COFFEE
11:00 – 11:30	<i>Sample environment and investigations under controlled humidity and gas atmospheres</i> Dirk Wallacher, Helmholtz-Zentrum Berlin
11:30 – 12:00	<i>TIPSI – time of flight instrument for powder diffraction, small angle scattering and imaging</i> Mogens Christensen, Aarhus University
12:00 – 13:10	Discussion II) in groups, specified topics, including summary afterwards
13:10 – 13:15	<i>Closing the meeting</i>
13:15 –	LUNCH

Discussion II

The discussion focus more on the scientific needs, then we know from all the presentations fairly well what's available when it comes to sample environment, what competences there are, and probably also something about future plans. We can discuss a "wish" list.

Moderator: Duncan Gregory, University of Glasgow

Appendix 3 List of participant

ESS Science symposia, April 26-27, 2012

Participant	E-mail	Affiliation
David, Bill	bill.david@stfc.ac.uk	ISIS, Rutherford
Hull, Steve	steve.hull@stfc.ac.uk	ISIS, Rutherford
Deen, Pascale	pascale.deen@esss.se	ESS, Lund
Henry, Paul	paul.henry@esss.se	ESS, Lund
Gregory, Duncan	Duncan.Gregory@glasgow.ac.uk	Glasgow University, Scotland
O'Hare, Dermot ^a	dermot.ohare@chem.ox.ac.uk	Oxford University, UK
Powell, Anthony	A.V.Powell@hw.ac.uk	Heriott-Watt, Scotland
Simonson, Mike	simonsonjm@ornl.gov	Oak Ridge National Laboratory
Hansen, Thomas ^b	hansen@ill.eu	ILL, Grenoble
Wallacher, Dirk	dirk.wallacher@helmholtz-berlin.de	Helmholz Zentrum Berlin
Boejesen, Espen Drath	espen@chem.au.dk	Aarhus University
Jensen, Kirsten	kirsten@chem.au.dk	Aarhus University
Christensen, Mogens	mch@chem.au.dk	Aarhus University
Eriksson, Sten	stene@chalmers.se	KB, CTH
Norberg, Stefan	stn@chalmers.se	KB, CTH
Rahman, Habib	habibur@chalmers.se	KB, CTH
Kinyanjui, Francis	kinyanju@chalmers.se	KB, CTH
Aktar, Sanjida	sanjida313@yahoo.com	BAEC, Dhaka
Hossain, Shahzad	shahzad_wossain@yahoo.com	BAEC, Dhaka
Karlsson, Maths	maths.karlsson@chalmers.se	Physics, CTH
Carlsson, Per-Anders	per-anders.carlsson@chalmers.se	KB, CTH
Gustafson, Johan	johan.gustafson@sljus.lu.se	LU, Lund
Eriksson, Lasse	lars.eriksson@mmk.su.se	Chemistry, SU
Häussermann, Ulrich	uhaussermann@gmail.com	Chemistry, SU
Jacas, Jordi	jordijacasb@hotmail.com	Chemistry, SU & ISIS
Andersson, Yvonne	yvonne.andersson@mkem.uu.se	Chemistry, UU
Edström, Kristina	Kristina.Edstrom@kemi.uu.se	Chemistry, UU
Gustafsson, Torbjörn	torbjorn.gustafsson@kemi.uu.se	Chemistry, UU
Roberts, Matthew	matthew.roberts@mkem.uu.se	Chemistry, UU
Rundlöf, Håkan	Hakan.Rundlof@kemi.uu.se	Chemistry, UU
Ångström, Jonas	jonas.angstrom@kemi.uu.se	Chemistry, UU



Group photo from the ESS Science Symposia at Chalmers



Steve Hull, ISIS, presenting *in-situ* studies on SOFC materials