TITLE: Enabling Neutron Investigation on Materials under Extreme Conditions

Type of activity: Joint Research Development

Leading beneficiary: EMFL (WP 1), ILL (WP 2), ESS (WP 3), PSI (WP 4), PSI (WP 5)

Partners: ESS, ILL, ISIS, LLB, MLZ, HZB, other
LNCMI Grenoble, LNCMI Toulouse, HZ Rossendorf, HMFL Nijmegen,
EPFLausanne, U. Edinburgh, U. Paris, U. Stockholm, KU, DTU

Abstract:

With its high penetration power in most materials and its sensitivity for magnetic as well as structural properties neutrons have been used for many investigations under extreme conditions. Nevertheless the geometrical requirements (methodological from the instrument design) as well as the sample size (imposed by the weak neutron source brilliance) have always lead to compromises in the sample environment. With the increased performance of instruments both at new (ESS) as well as existing sources - driven by new instruments and innovative neutron technologies - shifts those borders. Furthermore scientifically (high temperature superconductors, exotic electronic properties, quantum states) the drive is to ever-higher pressures and magnetic fields on ‘fine-tuned’ samples.

Since many years the application of high magnetic fields in neutron scattering experiments has been limited by superconducting magnets to vertical split pair arrangements up to 15 Tesla and to horizontal solenoid coils up to 20 Tesla. Higher magnetic field strength will be possible with new frontier developments in two directions. First, in a cooperation between the NSF partners, the European Magnetic Field Laboratories (EMFL) and an industrial partner (Oxford Instruments) a R&D study should be launched on **Stationary High Magnetic Fields up to 30 T** established by hybrid magnet coil sets using todays standard LTSC technique and 3rd generation of HTSC technology; this is proposed in **Work Package 1**.

Another path way is to use **Pulsed Magnetic Fields up to 50 (100) T** using standard low-resistive coil techniques (high conducting Cu-Ag alloy) cooled by liquid nitrogen at 77 K and powered by short-time Mega-Joule electric charge & re-charge devices. This is proposed in **Work Package 2** where again the cooperation with EMFL (i. e. the specialized labs in Dresden and Toulouse) is asked for.

High Pressure Techniques applied to materials in neutron scattering experiments also have to be rated as extreme conditions: pressure conditions in a range p = 1 – 100 GPa in combination with low and high temperature environments, simultaneously operated, are still exceptional physical sample

parameters. In **Work Package 3** we propose to continue the former successful collaborations between the SE experts at neutron centers (NMI3-FP7) and the Paris-Edinburgh high-pressure research community.

Neutron scattering at low and high temperatures including high magnetic field and high pressure typically results in an increased background signal due to extra construction demanded material in the beam path. It also results in serious restrictions on the available scattering configurations. In a conceptual effort, the neutron scattering facility experts should cooperate in order to define **Standards for Low Background Sample Environments.** According to the high demands on the construction details for the above mentioned state-of-the-art sample environment equipment, the selection of 'neutron-friendly' materials and the common development of in-situ sample manipulation devices are proposed in **Work Package 4**.

Though growing single crystal samples is typically the core business of the expert groups at universities, the large-scale facilities need to provide the essential knowledge especially in respect to the specific neutron requirements. This often translates into large samples of good crystalline quality besides the envisaged electronic properties. Standard laboratory equipment for magnetic characterisation at universities is unable to cope with the large crystals from neutron experiments. Moreover in-situ characterisation to relate the neutron measurement to the bulk property at a specific sample state is of increasing importance. Concerted actions towards crystal growth and (in-situ and ex-situ) sample characterization are port of **Work Package 5.**

Work Package 1: static magnetic fields

Univ. Partners

Estimated budget: TBD

Shifting the frontiers of the research on magnetic materials to a new state-of-the-art development it is envisaged to initiate a design study on a hybrid split-coil magnet system performing stationary magnetic fields fair above 20 T. Up today, this project is still under discussion and consideration among the experts in the four leading European High Magnetic Field Laboratories (EMFL) in Dresden (HLD, HZR), Nijmegen (HFML, RU/FUM), Grenoble (G-LNCMI, CNRS) and Toulouse (T-LNCMI, CNRS). The EMFL is focused on world leading developments of magnet technologies and to provide specific user experiments for scientific frontier research at the highest magnetic fields possible. In the past, the Grenoble lab and the Dresden lab shared hybrid magnet projects with the world leading industrial partner in the manufacture of superconducting (sc) cryomagnet equipment, Oxford Instruments (OI, UK). Following this perspective, we here propose to initiate a design study on a fully superconducting solution for a hybrid magnet coil system where the outer coils are manufactured in standard $NbSn_3$ technology and the inner coil set will be produced from newly developed high temperature sc-material. Given a positive outcome of the OI design and feasibility study, the collaboration between the industrial partner, the EMFL laboratories and the neutron facilities for producing and manufacture of such a hybrid magnet has to be decided and financially put forward.

Work Package 2: pulsed magnetic fields

Univ. Partners

Estimated budget: TBD

Given the outstanding ESS long-pulse neutron beam intensity, it is worth exploring todays pulsed high magnetic field techniques for applications to pulsed neutron scattering instrumentation. Thus, we propose collaboration between experts in neutron instrumentation and experts from the leading pulsed magnetic field laboratories in Dresden and Toulouse. The aim of this project is to define appropriate neutron scattering geometries with a favorable magnet environment structure (solenoid and split-pair coils). Pulsed magnetic field profiles with total duration of about 0.2 sec and peak values of 50 T (at ca. 50 msec) are envisaged; pulse repetition rates better than 100 sec should be pushed forward (for longer rep rates, max. peak field strength of 100 T are possible). High technology standards and safety aspects, as well, should carefully be envisaged in view on mobile electrical power stations, cooling by constant flow of liquid nitrogen and high voltage electromagnetic RF burst to be diminished below safe experimental and environmental limits.

Work Package 3: high pressure

Univ . Partners:

Estimated budget: TBD

As illustrated in the report from the Science Symposium 'Extreme Conditions' (http://europeanspallationsource.se/ess-science-symposia-schedule) performing neutron scattering experiments to investigate samples under extreme conditions is an important and growing area for many scientific communities. ESS will require state of the art equipment to achieve these - often combined - extreme conditions on a wide range of instruments. The instrument design must be compatible with the use of such equipment. For the most advanced extreme conditions, dedicated instruments are to be envisaged to respond to specific needs from the community.

Given that sample environment equipment for {T, B, E} is in many cases commercially available and could be purchased some two years ahead of the first operation phase, an emphasis in the current phase of the project is given on high pressure research. The wide range and diversity of pressure devices will be difficult to order off-the-shelf and requires R&D for the instruments and technical integration to be ready for user operation. During the construction phase 2014 - 2019, some of this work will be in-house prototype development mostly in partnership cooperation with neutron facilities ISIS, ILL, PSI, HZB, LLB and others. Extreme high pressure devices - like PE- & C-anvil cells - naturally will be pushed forward with the well–known expert groups (Paris, Edinburgh, Stockholm, others). Forming a high-pressure consortium will be important to attract funding for both the high-pressure equipment but also gathering (financial) support for a dedicated high-pressure instrument.

High pressure cells are containers or surrounding environments which provide the stability of load frames against the tremendous forces in order to pressurize the sample with gases, vapor and liquid or, forcing uniaxial pressure, applying piston-anvil techniques. In all pressure cell arrangements, the quest for extreme conditions limits in a range around 10 GPa, especially when simultaneously operated in combination with low (<1 K) and high temperature (>1000°C) environments and with high magnetic field. In the last years technically advances, especially on the diamond-anvil cell (DAC) technique, allowed neutron diffraction work close to 100 GPa (1 MegaBar) with crystalline sample sizes much below the former lower volume limit of 1 cm^3. We propose to continue the former successful collaborations between the SE experts at neutron centers (NMI3-FP7) and the Paris-Edinburgh high pressure research community (CSEC, U. Edinburgh and IMPMC, U. PMC. Paris). In view of the envisaged neutron spectroscopy instrument CAMEA at ESS, which is designed for optimal efficiency in the horizontal scattering plane, it is also proposed to develop a state-of-the-art high pressure cell for this instrument.

Work Package 4: materials for improved signal-to-noise

Estimated budget: TBD

The signal to noise ratio is the decisive factor over the success of a neutron scattering experiment. Cryostats, magnets and pressure cells and their combination are the most commonly used and demanded sample environment. The access to cryostats, cryomagnets, furnaces and high pressure devices usually results in an increased background signal due to a variety of materials in the beam path and restrictions in the available scattering configurations. Approaching this problem from the other side, one can maximize the signal by choosing the optimal sample size and containment and, in addition, fine-tune the scattering conditions utilizing in-situ sample manipulation. The possibility to realign the sample within a cold cryostat avoids time consuming warming and cooling procedures thereby increasing the efficiency of beam-time usage. Today, all proposing partners are working to some degree on these topics and there are many individual solutions to the same problem. We propose to undertake a collective effort to single-out the commonalities, explore the potential and technical limits of these solutions and develop new concepts for sample environment and work together towards solutions that can be shared amongst the facilities. This would lead towards a basic standard for sample environment equipment, which improves the accessibility and fosters the mobility of users between the European scattering facilities.

Work Package 5: Crystal Growth and characterization of superconducting samples

Partners: ESS (SAD), PSI (MKenzelmann), other

Estimated budget: 3FTE PhD, 15k€ travel, 75k€ equipment (furnace, ac susceptibility)

Though growing single crystal samples is typically the core business of the expert groups at universities, the large-scale facilities need to provide the essential knowledge especially in respect to the specific neutron requirements. This often translates into large samples of good crystalline quality besides the envisaged electronic properties. Standard laboratory equipment for magnetic characterisation at universities is unable to cope with the large crystals from neutron experiments. Moreover in-situ characterisation to relate the neutron measurement to the bulk property at a specific sample state is of increasing importance.

We here focus on equipment, which is robust and can easily be adapted to the in-situ purpose once ESS is up and running.

Preliminary work plan:

Year 1: PhD training activity, identification of required equipment, focus on flow growth

Year 2: Procurement and installation of equipment, experiments at existing RI, transfer of equipment to ESS

Year 3: scientific publications, final report

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