Perspectives for Neutron Science in Novel & Extreme Conditions

May, 27th - 31st, 2012, Zaragoza (Spain)
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INVITED TALKS
What Could a XtremeC Beamline Unveil in Materials Science?

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It is commonly accepted that experimental research under Extreme Conditions is mainly led by scientists having access to large facilities of synchrotron and neutron sources. The reason is probably due to the especially dedicated beamlines operating with beams (flux and energy range) and experimental setups that make them unique to explore the structure of condensed matter systems under the hard requirements imposed by sample environments under extreme conditions. Although beamstations incorporate the newest developments in high pressure, low/high temperature or high magnetic field, there are important novel phenomena in hard and soft condensed matter which are yet difficult to investigate with present experimental facilities due to limitations of sample volume, pressure cells or especial environments. Hence future developments in Extreme Conditions science must consider go beyond present instrumental limitations and at the same time offer an adequate infrastructure for users to do reliable and efficient experiments at large facilities. Furthermore, the possibility of conducting experiments at large facilities using either x-ray or neutron beams jointly with standard spectroscopic techniques (Raman, optical absorption, photoluminescence, etc.) is noteworthy as it provides a systematic in situ sample characterization in order to reproduce given experimental conditions. Furthermore this procedure gives a direct link between experimental results obtained at home laboratories or synchrotron facilities, with high-pressure experiments conducted in neutron facilities. In this way, developments in high volume, high pressure, and high magnetic field can be crucial to unravel a large variety of physical phenomena related to the interplay between spin state, electron-phonon coupling and orbital order governing the optical, magnetic and electrical properties, and that can be modified applying external pressure.

Spin transition phenomena occurring in pure or diluted transition-metal oxides like magnetite Fe₃O₄ [1], magnesiowüstite (Fe,Mg)O [2], multiferroic BiFeO₃ [3,4], delafossite (CuFeO₂) [5], low-spin cobaltite (La,Sr)CoO₂ [6] or high-spin CoF₃ or CsMnF₄ drive structural changes with pressure affecting their magnetic and electrical properties: high-spin (intermediate spin) to low-spin transitions; charge-transfer processes; insulating-metal transitions, etc. These systems illustrate how investigations using neutron probes under high pressure (30-40 GPa) and high magnetic field (20 T) can be crucial to reveal and eventually understand structural modifications and related phenomena yet unsolved in materials science. This is a contribution from the High Pressure & Spectroscopy Group, University of Cantabria.

References:
Neutron Diffraction in Pulsed High Magnetic Fields

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In high magnetic fields, various exotic states are induced. Magnetic order parameters and key characteristics of such phases can be examined precisely by neutron diffraction. To conduct neutron diffraction experiments in magnetic fields above 20 T, we have developed various types of pulsed field generators.

One of the most useful systems is a portable pulsed field generator for 30-40 T. It consists of mini-pulsed magnet, a compact capacitor bank and a cryostat insert. The insert equipped with the magnet can be put into a standard orange cryostat. The bank has the energy of 12 kJ only and thus can be installed with the space of 0.6 m³. Because of the compactness, experiments can be performed in most of neutron facilities over the world without modifications of spectrometers. To achieve higher fields, a larger capacitor bank is needed. For example, he magnetic field as high as 50 T was generated with a 250 kJ capacitor bank at J-Parc.

A data acquisition scheme is different between a reactor and a spallation sources.

(1) Reactor source
In reactor source, monochromatic neutron is used and a magnetic field dependence of a Bragg peak intensity is measured in each magnetic field pulse by time-dependent measurement. A typical number of field generation is 100-200 shots with the interval of 5 minutes at 30 T. Instead of a conventional time analyser system, a digital oscilloscope can be used to measure a time dependent histogram. Hence, no modification of the counter system is needed. Use of polarized neutron is also possible. We have examined the incommensurate-commensurate transition in Cr-spinel compound[1] and the successive phase transition of Tb boride.

(2) Spallation neutron source
In pulsed white neutron, Laue method can be used to scan a wide range in reciprocal space. The change of magnetic structure such as the shift of incommensurate peak can be traced efficiently[2]. Moreover, the number of field shot can be reduced for the strong instantaneous pulse intensity. For example, a strong magnetic peak can be observed in 20 shots at the 2nd target station of ISIS by using the strong enhancement in the cold neutron regime. We have examined the complex magnetic phase diagram of a multi-ferroic compound MnWO₄[2] at SNS.

In this talk the overview of experimental techniques and recent examples on neutron diffraction in pulsed magnetic fields are presented.

A capacitor bank installed in ISIS and the magnet with 33 mm outer diameter.

Acknowledgments:
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References:
The Road Ahead for the European Spallation Source

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With the political agreement in early summer 2009, to choose a site for the ESS, 17 European countries have joined together in a partnership to embark on a 3-year Pre-Construction Phase, including a full Design Update and Preparation to Build activities, prior to formally starting construction on the site in Lund in southern Scandinavia in 2013.

The ESS will be a 5MW long-pulse spallation neutron source and as such it will be a unique and uniquely powerful facility offering new opportunities for materials research using slow neutrons. Compared to the user’s experience today the ESS aims to offer measuring capabilities two orders of magnitude better than similar facilities today.

Our accelerator will be substantially superconducting using niobium for the cavities. Such metals suck up hydrogen from the environment and it is interesting that today the absorption of hydrogen by niobium is very relevant in accelerator component technology. Recent work on niobium cavities fabricated with ingot niobium carried out through a Brazil – India – Jefferson lab collaboration has indicated major advances in performance of such cavities. After proper verification such an advance could well prove advantageous for the performance and reliability of ESS.

Planning for a new 1.5 B€ scientific facility which will be operational a decade from now requires a new approach. For example it is likely by 2020 that the way in which researchers interact with central science facilities will have changed dramatically thanks to the very rapid advance of robotics, computing and IT methods. Equally well, the energy consumption of big facilities puts a significant burden on the annual operating budget of such facilities and novel methods must be found to manage the energy inventory of the ESS. One consequence of the high demand for energy is the environmental impact, which has political overtones. ESS has pioneered an innovative strategy for its energy inventory, which envisages using renewable sources, recycling waste heat and being especially vigilant with energy use and conservation. This not only reduces our costs but it represents a responsible approach to our environmental footprint. Our plan is to be CO₂ neutral over the lifetime of the facility.

ESS will be built on a truly green field site. Green field in the psychological as well as the physical sense. This gives an opportunity to revisit the standard methods of dealing with the user community, providing facilities, which will give added value to the visiting researcher and those who remain at their home laboratories. Thanks to the construction of the high brightness synchrotron source MAX IV being built in the same location as ESS mutual advantages by bringing the user communities of the two sources together will be a gain for all. A seventeen hectare piece of land between the two facilities will be used to build INXS, the institute for neutron and x-ray science which will incorporate facilities for users such as meeting places and conference rooms but, more importantly, an assembly of separately identifiable laboratories similar in nature to the Partnership for Structural Biology which was so innovative on the ILL-ESRF site in Grenoble. Similar centres for soft condensed matter, material under extreme conditions and material for climate, energy and health will be explored. The changing situation with neutron detector materials supply has refocused the development of neutron detectors away from gas detectors using helium-3 to solid detectors using boron-10. This is a challenge for the community which is being addressed energetically. I will attempt to address these questions and give a personal view of future developments.
Some Open Question and Perspectives in the Physics of Solids at Very Low Temperatures

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Temperature measures the average energy of a material above its ground state. In Nature, temperature ranges from millions of Kelvin at the centre of stars to 2.7 K, the characteristic temperature of the “background radiation” arising from the remains of the Big Bang. This very wide range, perhaps comparable only to scales of time, has been substantially enlarged by human science. Experimental physics has explored the behaviour of matter at temperature only a few nanoKelvin above absolute zero. Experiments at very low temperatures have enabled to explore the ground state of materials and the emergence of phenomena such as superfluidity, superconductivity and exotic magnetic phases of electronic as well as nuclear spins. In this talk, I review examples that show how neutron scattering techniques contributed to the current understanding of some of these phenomena and illustrate, with examples, a few of the open questions and perspectives in this field.
Protein Crystallography: What Can Nuclear Polarisation Do For Us?

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Neutron diffraction on samples with large hydrogen content, especially large organic and protein samples, generally suffers from a strong featureless background due to strong incoherent scattering from the protons. This is particularly evident in the two-dimensional projection of Laue diffraction, a technique which is otherwise undergoing a renaissance thanks to the development of large-solid-angle image-plate detectors, notably on LADI [1] and VIVALDI [2] at the ILL, and most recently on KOALA at the OPAL reactor at ANSTO. Despite the strong incoherent background, the larger coherent neutron scattering length of hydrogen relative to other elements compared with the situation in X-ray diffraction more readily yields answers to specific questions on, e.g. protonation states, hydrogen positions, and dynamic disorder of hydrogen. Deuteration can be used to reduce the incoherent background, but sample growth may be difficult or even impossible, and there may be an isotopic difference between the deuterated and non-deuterated structures. An intriguing alternative is parallel polarisation of the incident neutron beam and the nuclear spins of the hydrogen atoms [3].

Achieving nuclear polarization is quite challenging technically. It has been exploited rather successfully in small-angle neutron scattering from solutions [4,5], but rarely in protein crystallography, and never in Laue diffraction. A recent polarised-neutron Laue diffraction experiment on a single crystal of Nd-doped La₂Mg₃(NO₃)₁₂.24H₂O with the proton spins aligned by dynamic nuclear polarization has demonstrated however that the incoherent background is indeed reduced and also that the intensities of the Laue reflections can be enhanced or diminished significantly to give a form of contrast variation on one and the same sample [6].

The opportunities and current limitations of nuclear polarization in neutron protein crystallography will be presented and critically reviewed against the present capabilities of crystallography on non-polarised hydrogenous and deuterated samples.

References:
Mineralogy at High Pressure and High Temperature

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The challenges associated with understanding the behaviour of materials in the interior of planet Earth hinge on understanding the response of minerals and aggregates of minerals to the extremes of pressure and temperature experienced at depth. How the Earth evolved in its early history, and how it works today in terms of the generation of magnetic field, of seismic stresses and of volcanic hazards, are all problems whose solutions lie in interpreting high P/T mineral behaviour. Phase transformations involving small rearrangements of atoms between solid state polymorphs at the Å scale drive major tectonic processes on a vast continental scale. Such transitions control the dynamics of matter and heat flux in the planet’s interior and may generate deep Earthquakes, as well controlling the patterns of thermal convection from depths as far as 3000 km below the surface. The nature of the material forming the deeper reaches of the planet remains a topic of debate, stymied by a lack of reproducible data.

What is known is that phase transitions between solids as well as changes of state dominate much of what is seen in geophysical observations. Melting in the shallower reaches of the silicate mantle generates new crust at rates that seem to have varied through time and may link to variations in global climate and in species diversity. Crystallisation of the inner core within the molten outer core may also be a dominant contribution to heat flow within the planet, and to the balance of elements throughout the Earth. It is known that small amounts of light elements, in particular hydrogen, can significantly alter mineral properties and stability at high pressure and temperature. It seems obvious that neutron scattering should play an important role in aiding our understanding of light element fluxes (in particular hydrogen, or [experimentally] deuterium), sources and sinks within the Earth. Many of the phases of interest contain small amounts of hydrogen in the presence of heavy elements, and prove a challenge for X-ray methods. The ability to probe molecular dynamics of hydrous species and carbonate species in silicates and other oxides makes neutron experiments very attractive to the mineralogist.

The ability to scatter from magnetic moments is a further boon provided by neutron methods, and the magnetic behaviour of minerals at depth within the Earth and planets such as Mars is a topic of key interest. The challenges of applying neutron methods to such problems, however, are almost all associated with the small volumes and large apparatus typically associated with high pressure sample environments. The penetration of neutrons in larger samples may also prove useful for understanding the mechanical, rheological, and viscoelastic properties of Earth materials at the conditions of their formation, deep in the roots of mountain systems.

I shall outline some current problems in Earth materials for which neutron methods appear well adapted, and pose the questions that must be answered technically if such studies are to be successful. These questions largely focus on the challenges of carrying out high-pressure and high-temperature science on relatively small samples in complex sample environments. Some challenges have been successfully addressed in recent years, others remain before us.
Levitation of Liquid Metals in Space and on Earth

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The knowledge of thermophysical properties of liquid metals and their liquid dynamics is of fundamental importance for the understanding of their liquid state. Thermophysical properties like viscosity and surface tension are an essential input for large-scale simulations in materials design. In order to measure thermophysical and microscopic properties in the liquid state accurately, levitation techniques have been developed. Electromagnetic levitation is applied in several ground based and microgravity experiments like parabolic flights, sounding rockets, and the International Space Station. In electrostatic levitation, positioning and heating are decoupled and, hence, also non-conducting materials can be processed. Electrostatic and electromagnetic levitation can be used with various diagnostic methods: Direct imaging allows for the determination of the liquid density, while surface tension and viscosity are measured by monitoring the induced oscillations of a levitated droplet both on ground and under microgravity.

Experimental investigations on refractory metals suffer from the chemical reactivity of the melts at the high temperatures involved. This circumstance poses a major challenge especially in scattering experiments where the choice of a proper sample container material is already limited by suitable scattering and absorption cross sections. For this reason experiments on melt structure and atomic dynamics in these systems are rare, despite of their fascinating importance in industrial processes. In recent years much progress has been achieved through container-less processing of the sample droplets by the use of levitation techniques in-situ on scattering instruments. The challenges for such measurements are a stable positioning of a large sample droplet and a rather homogeneous sample temperature.

We apply both neutron scattering and synchrotron radiation to investigate structure and dynamics of liquid metals at the atomic level. E.g. recently, the field of liquid diffusion experiments advanced through the use of quasielastic neutron scattering on levitated metallic droplets for accurate measurements of self-diffusion coefficients in high temperature metallic liquids. In this presentation advances in electromagnetic and electrostatic levitation for the application on neutron scattering instruments will be presented. Recent experimental results are discussed in the context of the relation of self-diffusion and viscosity, as well as the relation of properties of mass transport and the atomic melt structure.

References:
Spin Fluctuations and Lifshitz Transition in Ferromagnetic Superconductor UGe$_2$ Probed by Larmor Neutron Diffraction Under Pressure.

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We present high resolution measurements of the lattice constants of UGe$_2$ under pressure probed by a novel technique that utilizes Larmor precession of polarized neutrons to surpass the resolution of conventional scattering methods by an order of magnitude. At low temperature UGe$_2$ is ferromagnetic up to critical pressure $p_c$ but superconductivity is peaked at a lower pressure $p_x$ coinciding with a less understood transition within the ferromagnetic state [1]. At ambient pressure we observed sharp anomalies in the lattice parameters at the Curie temperature, $T_{Curie}$. At higher pressure sharp anomalies in the lattice parameters at both $T_{Curie}$ and $T_x$ (the characteristic temperature for the transition that occurs at $p_x$) shift to lower temperatures in agreement with the known phase diagram.

We show that the electronic part of the thermal expansion is dominated by the contribution due to the ordered moment in accord with the spin fluctuation theory at $p<p_x$, however at $p> p_x$ we identify an additional contribution to the thermal expansion due to fluctuating moment associated with the metamagnetic transition at $T_x$. At pressure near $p_x$ and temperatures above $T_{Curie}$ we observed positive contribution to the thermal expansion along the a and c axes of the orthorhombic structure of UGe$_2$, most likely originating from the pressure driven Lifshitz transition[2] in the quasi-two-dimensional Fermi surface. We discuss the role of the Lifshitz transition and a plausible Kondo lattice scenario in stabilising superconductivity in ferromagnetic state.

References:

Sample Changers at Low Temperature: Present Status and Perspectives

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The powder diffractometer, POWGEN, at the Spallation Neutron Source – Oak Ridge National Laboratory has been successfully using the Fast Exchange Refrigerator for Neutron Scattering (FERNS), a low temperature sample changer since 2010. FERNS was built as part of a Small Business Innovation Research project for use on POWGEN. It can hold up to 24 specially designed sample filled vanadium cans on a carousel and loads samples using a bayonet style mechanism. FERNS can operate with a sample position temperature range of 10K to 320K, can cool from 300K to 10K in 45 minutes, and can successfully change samples at 10K in less than 20 minutes. FERNS is a first generation low temperature sample changer that has been used for the majority of experiments on POWGEN.
Neutron Science at High Temperature: Tendencies and Perspectives

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Studies of the liquid state present an obvious fundamental interest and are also important for technological applications since the molten state is an essential stage in various industrial processes (e.g. glass making, single crystal growing, iron and steel making).

Most of the physical properties of a high-temperature liquid are related to its atomic structure. Thus it is important to develop devices to probe the local environment of the atoms in the sample. At very high temperature, it is difficult to use conventional furnaces, which present several problems. In particular, physical contact with the container can contaminate the sample and/or modify its structural properties. Such problems encouraged the development of containerless techniques, which are powerful tools to study high-temperature melts. By eliminating completely any contact between sample and container, it is possible to study the sample with a very high degree of control and to access very high temperatures. An additional advantage of levitation methods is that it is possible to supercool hot liquids down to several hundred degrees below their equilibrium freezing point, since heterogeneous nucleation processes are suppressed.

Various containerless techniques have been applied to determine physical properties of levitated liquids [1]. Some of them have been combined with neutron scattering techniques to study the structure and the dynamics of molten materials. We will give an overview of the levitation techniques that have been used at neutron sources, particularly at ILL. We will focus on the various developments that we have made with aerodynamic levitation as collaboration between ILL and the CEMHTI in Orléans (France).

Acknowledgments:
We are grateful to the staff members, technical and scientific, at ILL and CEMHTI for their technical help and assistance during the experiments and/or the design of some of the experimental setups.

References:
A New Type of Pressure Cell for Neutron Experiments

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In the first part of this talk we will present a review of large volume pressure cells used over the last decades in neutron experiments under high pressure, specifically the Paris-Edinburgh1 cell and the so called Kurchatov-LLB2 pressure cell.

In the second part we will make a description of a new pressure cell for neutron diffraction experiments for small samples with volume of 1 mm$^3$ and pressures up to 30 GPa. This cell of a titanium alloy can be used with sapphire, moissanite (SiC) and transparent synthetic diamond anvils. This device has been designed and optimized using finite element calculations. The pressure is measured by the luminescence of ruby3. We built a small scale prototype that has allowed us to verify its performance under pulsed magnetic fields up to 60T and temperatures up to 2K4. This type of cells has the advantage of allowing \textit{in-situ} optical characterization (absorption, photoluminescence, Raman) of the samples in the setup of neutron scattering.

References:


Neutron Scattering in the Mbar Range?

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In my talk I will review the technical challenges and perspectives for neutron scattering in the Megabar range. I hope my contribution will give useful recommendations on what kind of developments and investments are needed to achieve this, and maintain Europe competitive in high pressure neutron scattering in general.
Neutron scattering from planetary materials

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In the context of the outer solar system, the ices (methane, ammonia and water) form the basis of mineralogy. The planets Neptune and Uranus both have mantles containing significant fractions of these ices at high pressures and temperatures. Convection within these mantles is believed to be the cause of the non-axial non-dipolar magnetic fields of these planets. Models of this process rely on information on the behaviour of the ices at high pressure and temperature, for example, densities, speciation, viscosity and thermal- and electrical-conductivity. Information on these crucial properties has been difficult to obtain under the appropriate P-T conditions.

In my talk I will give examples of how high pressure studies can provide the microscopic basis for macroscopic models and I will review prospects for developments in the future.
Metal organic frameworks (MOFs) are crystalline materials that contain metal-ions or metal-ion clusters as nodes and organic ligands as linkers to form 1-, 2-, and 3-D structures. Their structural versatility and multifunctional properties have sparked much interest in advanced materials synthesis. Due to their modular nature, many of these materials can be constructed by design. Over the last decade there are several MOFs that reportedly have high surface areas allowing them to physically adsorb significant amounts of gas. Adsorption of molecules in functionalized and high surface area microporous materials is of technological importance in a multitude of areas ranging from catalysis, drug delivery, chemical separations and energy storage to personal care products. Through careful selection of the ligand and metal, which control pore size/shape and MOF-adsorbate interactions, their uptake properties can be tuned. Over the past several years we have focused our research efforts on understanding the properties of hydrogen interactions within a variety of microporous materials with the goal of improving new hydrogen storage materials.

One of the key stumbling blocks on the road to commercialization of hydrogen-powered vehicles is the current lack of a suitable storage medium that significantly improves on high-pressure gas tanks. Several classes of materials have been studied exhibiting a range of hydrogen retention and release properties from the weakly interacting gas adsorption systems to those containing strong chemical bonds. While steady progress is being made in material synthesis, parallel work in understanding the underlying physics and basic properties of these materials that lead to attractive hydrogen storage properties is invaluable in directing current efforts. I will describe our recent and on-going research activities that combine structural studies with dynamics and *ab-initio* calculation that together provide a complete picture of these potential hydrogen storage materials along with adsorption processes of other gases.

References:


The Soft Approach: Fibre Diffraction Under Controlled H/D Humidities

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The importance of neutron scattering for soft matter dynamics goes without saying, and sample environment has played a key role. This has been especially the case in structural studies, more so than in measuring dynamics. I will give a series of examples of research from the soft matter community which shows how sample environment, sometimes quite adventurous, has enabled new science. These examples will demonstrate current capabilities. In building the next generation of neutron instruments at the ESS, I will discuss what the soft matter community might need for the future.
Neutrons have a leading role in the studies of a large variety of Soft Materials (physical chemistry or bio-inspired systems). This is foremost due to the possibility of labelling by deuteration, which allows highlighting the parts of interest in the multi-component systems common in soft matter. Both the structure and dynamics of soft materials are ideally elucidated by Small Angle (SANS), Reflectivity (NR) and Quasi-elastic Neutron Scattering due to the characteristics time and length scales probed by these techniques (from picoseconds to few hundred nanoseconds and from fraction of nanometers up to several hundreds of nanometers). In addition, neutrons are non-destructive and their intrinsic properties allow them to easily penetrate matter (contrary to electrons and x-rays), which allows developing complex sample environments without parasitic scattering or significant loss of scattering signal from the sample.

Most soft materials are very sensitive to external stimuli: deformation, electric or magnetic fields, UV or visible irradiations, pH, concentration, humidity, temperature and pressure variations, etc.. More kinetics and dynamics studies using advanced in-situ devices need to be developed to exploit efficiently the existing and foreseen high flux sources like the European Spallation Source. Present environments require large sample volumes that limit technical performances of high pressure, T-jump or shear devices. Furthermore, many experiments are today inaccessible due to the limited quantity of sample available (labelled or biological molecules). Great advances in our understanding of soft materials properties can be achieved by pushing forward the limits of preparing samples, applying external stimuli and recording in-situ/in-operation experimental data.

In the last ten years, a clear tendency of research has focused on bio-inspired materials. Neutron techniques, especially NR, have recently started to offer high performance tools for the study of biological membranes. A great challenge in the field is to succeed in standard production of model membranes, of recipes for specific deuteration and in the development of novel modelling methodology, including molecular scale information from MD simulations.

In order to make the best use of neutron scattering for soft and bio materials studies, the Work Package “Advanced Neutron tools for Soft and Bio materials” of the NMI3-II FP7 project will focus on:

1. A platform for model biological membranes,
2. Stopped-flow, Pressure and electric field cells, Multi-angle light-scattering combined to SANS
3. Temperature controlled humidity chambers,
4. Cryogen-free cryostat featuring sample changer

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Neutron Scattering Studies of Quantum Dimer Magnets Under Extreme Conditions

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Neutron scattering is a key experimental tool for the study of the exotic ground states and characteristic excitations in strongly correlated electron systems, quantum magnets and related materials of current interest. Quantum phase transitions in such systems may be realized by the application of high magnetic fields or hydrostatic pressure at ultra-low temperatures. Examples are presented for quantum phases transitions studied by neutron scattering under such extreme conditions, and will be discussed in the context of current limitations and future perspectives for such experiments.
High Magnetic Field and Low Temperature Sample Environments: Perspectives for Neutron Scattering

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A summary of Oxford Instruments activities to develop novel cryogenic and cryogen-free technologies for low and ultra-low temperatures and for high magnetic fields, and the application of such technologies to the needs of neutron scattering facilities.

Over several years continuous progress has been made in the design of complex high field magnets for beamline applications, in many cases through close collaboration with neutron scientists, and with the neutron facilities’ sample environment leaders. Magnet designs ranging from ultra-high field SANS system to custom engineered wide-angle scattering systems have been developed. A full choice of cooling options will be described, from cryogenic to recondensing to purely cryogen-free, and the relative advantages of each explained.

An active development programme to extend the upper field limits of purely superconducting magnets will be described. Using HTc insert coils and enhanced LTc wire for the outsert coils, then we envisage fields of >30T being available within a few years time.

Finally, a state-of-the-art cryogen-free dilution refrigerator, with fast sample change options, and accelerated cooling technology for cooling large magnets or other masses such as pressure-cells has been developed.
Magnetic Neutron Scattering Under Extreme Conditions

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Neutron diffraction experiments often play the crucial role in solving various physical problems. Due to its interaction with both, nuclei and open electron shells, neutrons can provide information on the crystal and magnetic structures. As the magnetic state often depends strongly on external conditions, studies of the magnetic state on external variables are very common. These include for instance the determination of magnetic structures and phase transitions as a function of temperature, magnetic field, pressure and other thermodynamical conditions like electric field and construction of phase diagrams. Very important ingredient is the fact that neutron beam is a weak probe that is able to penetrate certain materials rather easily without substantial loss of intensity. This enables construction of complicated sample environments and combination of low temperatures down to a mK range with high magnetic fields and pressures, leading to truly multi-extreme conditions. Under such conditions one can study for instance quantum critical phenomena or studies of coexistence of superconductivity and long-range magnetic order.

In the contribution we describe existing, more or less standard sample environment used around the world and briefly present very recent project that combines dedicated neutron scattering instrument with a horizontal solenoid magnet with tapered cones. This project is being realized at Helmholtz-Zentrum Berlin in collaboration with the National High Magnetic Field Laboratory of Florida State University, Tallahassee, FL, USA. The magnet utilizes hybrid (resistive insert and superconducting outsert) technology and is capable to produce static magnetic field of 26-32 T, depending on the power that is between 4 and 8 MW (see also contribution by P. Smeibidl). The dedicated instrument is of time-of-flight (TOF) type and optimized for diffraction at extreme conditions.

In the contribution we further discuss few recent scientific activities in the field of neutron diffraction performed at various institutions (e.g. at HZB, ILL, PSI) on frustrated magnetic systems, iron based superconductors and experiments under combined extreme conditions, discuss existing limitations and drawbacks and outline our future scientific prospects using the extreme sample environment conditions.

References:
The Hybrid Magnet for Neutron Scattering at Helmholtz Centre Berlin

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The Helmholtz Centre Berlin (HZB) is a user facility for the study of structure and dynamics with neutrons and synchrotron radiation with special emphasis on experiments under extreme conditions. Neutron scattering is uniquely suited to study magnetic properties on a microscopic length scale because neutrons have comparable wavelengths and, due to their magnetic moment, they interact with the atomic magnetic moments. Many materials and problems of outstanding scientific importance, including field induced normal state in high-TC superconductors, lie beyond the 15 T technological barrier for the existing split-coil superconducting magnets (see talk of K. Prokes for more details on scientific opportunities). To open up higher fields to neutron research requires a reinvented approach with fundamentally different magnet technology and neutron instrumentation.

At HZB a dedicated instrument for neutron scattering at extreme magnetic fields and low temperatures is under construction, the Extreme Environment Diffractometer ExED. It is projected according to the time-of-flight principle and for the special geometric constraints of analysing samples in a high field magnet. The aim is the construction of a multi purpose instrument which offers diffraction experiments as well as small angle neutron scattering and inelastic scattering.

Following our past experience only steady state fields are adequate to achieve the goals of this project. In particular inelastic scattering studies, which proved in the past to be most rewarding, would virtually be excluded when using pulsed magnets. The new hybrid magnet, designed and constructed in collaboration with NHMFL, Tallahassee, will not only allow for novel experiments, it will be at the forefront of development in magnet technology itself. With a set consisting of a superconducting cable-in-conduit coil and different resistive coils maximum fields above 30 T will be possible with cooling power up to 8 MW for the resistive part. To compromise between the needs of the magnet design for highest fields and the concept of the neutron instrument, the magnetic field will be generated by means of a coned, resistive inner solenoid and a superconducting outer solenoid with horizontal field orientation. The installation of the necessary technical infrastructure for magnet operation, 20 kA power supply, water cooling system and Helium refrigerator, which is extensive compared to the magnet systems currently under use, needs a separate building beside the Neutron Guide Hall with the neutron instrument and the magnet system.

To allow for experiments down to Millikelvin temperatures the installation of a 3He or a dilution cryostat with a closed cycle precooling stage is foreseen.
Control of AFM Domains and Polar Domains With Electric and Magnetic Fields in Multiferroics

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In the last decade, the discovery of large magnetoelectric coupling in bulk transition metal oxides has triggered intense research in the field of multiferroics. The control of ferroelectric domains with magnetic field and, conversely of magnetic domains with electric field, has been achieved in many systems, for some of them at room temperature. Neutron scattering has played a major role in revealing the complex magnetic structures of these systems, but also in determining the AFM domains populations by polarimetry measurements. We will review some of the experiments that can be done currently, and discuss the need for high magnetic fields.
The NHMFL is one of the leading organizations worldwide in the development of high-field magnet systems, presently operating a 25 T split magnet, a 45 T hybrid, and a 100 T pulsed magnet. The status of magnet systems suitable for neutron scattering is presented.

The latest concepts rely on YBCO high-temperature superconducting tape and have been developed in collaboration with HZB and ESS. Conceptual designs of two configurations have been developed: 1) a 25 T vertical split and 2) a 30 T double-conical, horizontal-bore system. Both magnets rely heavily on the YBCO tape-based magnet technology being developed at the NHMFL for use in a 32 T magnet which is expected to be operational in 2013.

A 25 T double-conical hybrid magnet is being developed for the Helmholtz Zentrum Berlin. This magnet will feature a 50-mm horizontal bore with converging-diverging cones to allow neutron spectroscopy experiments. The largest sub-system in the magnet is the outer superconducting coil which employs Cable-In-Conduit Conductor technology. All of the conductors for the magnet have been fabricated and delivered to Tallahassee. Winding of the coil is nearing completion as of this writing and might be complete by the time of the conference. The cryostat for the coil has been ordered. Design of the resistive insert coils is underway.

The recently completed 25 T split magnet uses resistive magnet technology to provide up to 25 T in a 32 mm bore with four ports at the mid-plane, each of which includes an 11.4° vertical scattering angle and a 45° horizontal scattering angle. The magnet reached 25 T in July 2011 and has been used in various visible-optics spectroscopy experiments to date. To complete this magnet system, a new technology: Split Florida Helix was developed. The magnet can be operated with field either vertical or horizontal and we intend to build a second set of inner coils in coming years that will provide a larger gap with reduced field to allow a cryostat to be installed perpendicular to the field. The dc power requirement for the magnet is 28 MW.
Helix Development for High Field Magnets

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Few facilities worldwide have the mission to offer access to magnetic field well in excess of what is obtained by commercial low temperature superconducting magnets. The Laboratoire National des Champs Magnétiques Intenses (LNCMI) is one of them and provide access to searchers to magnetic fields up to 35 T using copper based magnets (with an “hybrid project to reach 43 T) at the Grenoble site and 80 T (with the aim of reaching 100 T) on the pulsed field installation located in Toulouse.

In addition to the classical “Bitter plate” technology, the LNCMI has developed the helix technology for high field magnet development. In this technology the winding of the coil is directly obtained by cutting copper alloy tubes in helical shape. We will give an overview of the high field magnet development based on this technology and explain the interest of this technology for the production of pulsed magnetic field with enhanced duty cycle as required for combined use with neutrons. In addition an overview of the high temperature superconducting developments at the LNCMI will be given.
ORAL PRESENTATIONS
Processes in Engineering Materials Under Extreme Thermal and Mechanical Loads

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Ever increasing demand for better performing engineering materials pushes material engineers to turn to multiphase and/or multicomponent materials and advanced processing methods. Since properties of such materials depend heavily on their microstructure created during processing, understanding and control of the processes responsible for microstructure evolution has become vital in modern material engineering research (Material by Design). Microstructure forming processes active during industrial processing of materials (e.g. forging, welding, spark plasma sintering, combustion synthesis) frequently take place under extreme thermal and mechanical loading conditions (high temperatures, complex loads and histories, extreme heating/cooling rates, large loading rates). It is challenging to follow them by in-situ methods and use the results to improve numerical models of industrial processing.

On the nanoscale, new opportunities have been recently opened thanks to the development of new transmission electron microscopy technique with simultaneous ultrahigh spatial and time resolution /4D TEM/ [1]). With this technique, for example, atomic motions during martensitic transformation can be followed with picosecond time resolution.

On the mesoscale, it is relatively easy to follow fast evolution of some physical properties of materials processed in laboratory conditions with very high time resolution (e.g. following stress, strain, electric, magnetic properties). Recently, technological developments of 2D CCD X-ray detector cameras opened new possibilities to use diffraction of energetic synchrotron X-rays to follow fast evolution of microstructures, stresses and phase fractions in processed materials with time resolution approaching ~1 ms. As a case example, results of simultaneous evaluation of force, electric resistance and synchrotron X-ray diffraction during short pulse electric current heating of thin metallic filaments [2] will be briefly discussed.

Really challenging, however, would be to follow the fast processes in engineering materials during its full scale industrial processing. Engineers frequently use for this purpose Gleeble Simulators [3] allowing for application of complex fast thermomechanical loadings to small material samples which simulate real conditions the material is exposed to during full scale industrial processing. Main goal of Physical Simulations is to develop new processing technology in laboratory scale and transfer it afterwards to the full scale industrial process.

Engineering materials exposed to simple thermomechanical loadings have been successfully investigated by in-situ TOF neutron diffraction methods on dedicated engineering diffractometers at neutron spallation sources (e.g. ENGIN-X, SMARTS, VULCAN). Longer acquisition times of neutron radiation and lack of dedicated equipment for Physical Simulations at neutron diffraction beam lines are currently two major factors limiting the possibility to perform Physical Simulations with neutrons. Science concept of Complex-Environment Engineering Diffractometer /CEED/ [4] proposed for European Spallation Source /ESS/, which attempts to overcome these two limitations will be briefly introduced.

References:
Sample Environment for Engineering Materials Research:
from Robotic Texture Analysis to \textit{in situ} Friction Stir Welding

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The availability of sophisticated sample environments is an essential requirement for the success of many scattering experiments. This is also true for experiments in the field of engineering sciences. Typical examples for standard engineering experiments are texture analysis, residual stress analysis, or phase analysis. Such experiments are performed, e.g., at high temperatures or under external mechanical loads. However, there is much room for developments going beyond the standard experiments.

In texture analyses, sample throughput often is a problem when engineers want to look at large sample series resulting from the requirements e.g. of process optimization. This problem can be solved with a novel robotic sample changer, as it is already available at STRESS-SPEC at the FRM II \cite{1}. Such a six-axes-robot cannot only be used for sample changing, but also for positioning and rotation of a sample, overcoming the spatial limitations of a Euler cradle. In general, such robotic technology can also be used for sample positioning, sample changing, or sample environment changing for other type of experiments.

Dilatometers are commercial instruments for the study of phase transformations as a function of temperature measuring the extension of a sample, which are available in many scientific and industrial labs. It can also be equipped with a deformation unit for modeling deformation steps at different temperatures. HZG is running such a dilatometer including deformation and differential scanning calorimetry units at a synchrotron beamline for user access with great success. It is planned also to modify such a commercial dilatometer for use at a neutron beamline. Pulsed sources are ideally suited for this, because a limitation in the range of scattering angles is possible.

A strong, pulsed neutron source also offers the possibility for the \textit{in situ} study of the friction stir welding process. A large machine is required for this to apply the necessary welding forces, making such a sample environment a rather extreme one. Only the time-of-flight technique enables the limitation of scattering angles that cannot be avoided in such a case. In contrast to an existing synchrotron experiment using high X-ray energies, the development of residual stresses could easily be studied during friction stir welding using neutrons because the wavelength range enables a scattering angle of 90°.

Examples for the three mentioned sample environments will be presented and future possibilities in combination with an engineering diffractometer at the ESS will be highlighted.

References:
Vortex Depinning Studies with SANS at Ultra Low Temperatures

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Small angle neutron scattering (SANS) was proven to be a versatile tool to study the vortex lattice and the flux line structure of superconductors. The symmetry of the vortex lattice can be used to reveal unconventional order parameter symmetries or strong anisotropies in the underlying Fermi surface. Moreover, multi-domain behaviour and to some extend pinning related disorder can also be observed. In order to study dynamic processes of a vortex lattice we built a modified setup, which allowed us to run electric currents through the sample while observing the lattice with SANS. With this setup we were able to observe the transition from a static vortex lattice to a freely flowing lattice. To our knowledge, this is the first experiment of this kind, which was performed in the sub-Kelvin temperature range and in magnetic fields of a few Tesla.

We will present current dependent SANS measurements on a heavy fermion superconductor at very low temperatures. We will discuss the technical challenges that we encountered and show how they were solved.
Large Volume Pressure Cell for Inelastic Neutron Scattering Spectroscopy

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The new interest and demand of the biophysical community, interested in food science and biology under extreme conditions, has pushed to the development and construction of a pressure samples holder adapted to large volume solution (0.7 cm\(^3\)) and suitable for neutron scattering. The sample holder, made in titanium alloy, is designed to reach the 3 Kbar but can be improved to go up to 7 Kbar. The cell design is also compatible to be used into most of ILL Orange cryostats, for Pressure vs Temperature studies, and mounted on a sample holder stick.

The pressure device has been conceived with a flat geometry (Figure 1). It is constituted of two manufactured titanium pieces which are hold together with height 12.9 security class screws and thermal treated aluminium joint. The illuminated neutron window has an outer diameter of 27.8 mm and an inner diameter, the sample illuminated part, equal to 19.5 mm.

The total thickness of the two illuminated titanium windows is of 11 mm, which brings to a calculated transmission, of the empty cell, equal to 0.52. The cell is conceived for in situ measurement. The hydrostatic pressure is achieved through a hand pump and monitored through a pressure sensor mounted on the cell. The loading, unloading and cleaning of the cell can be performed, without opening the device, throughout a set of capillaries connected at the pump. The actual thickness of the sample solution has been adapted to the new Brillouin Spectrometer BRISP, at ILL, and it is of 6.7 mm. In order to take into account the multiple scattering, arising from the important sample thickness, a program correction is under development. The performance of the device is illustrated with preliminary results of inelastic neutron experiments on high concentrated protein solution.

\(\text{Figure 1. Large volume and flat geometry pressure cell.}\)
Nuclear Spin Polarization of Nd by the Electronic Field in the Range of mK

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The alignment of nuclear magnetic moments is difficult to observe by neutron diffraction since it requires very high magnetic fields and very low temperatures. Moreover, the nuclear magnetic moment is three orders of magnitude smaller than the electronic one. Nevertheless the electronic spins create a hyperfine magnetic field much stronger than the fields available in a laboratory. The resulting nuclear polarization leads to some interesting phenomena that can be investigated by neutron diffraction. A cause of the incoherent scattering of neutrons is the thermal distribution of different nuclear spin states. As temperature decreases, they become aligned by the electronic field and some incoherent scattering becomes gradually coherent. The spatial periodicity of the ensuing nuclear order is the same as that of the electronic moments but the physical origin of the scattering is the interaction between the neutron and nucleus. The result is that the intensity of the usually considered “magnetic” (electronic) Bragg reflections increases. New reflections also appear because those with \( q \) parallel to the magnetic moment are allowed for the nuclear scattering.

\(^{143}\text{Nd}\) and \(^{145}\text{Nd}\) are especially adequate for studying this phenomenon because of the following reasons: 1) they possess high incoherent scattering lengths, 2) their nuclear magnetic moment is also relatively large, thus easily polarizable at temperatures above 100 mK, and 3) the hyperfine field can be controlled by the choice of material since the Nd electronic moment strongly depends (between 1 and 3.27 \( \mu_B \)) on the local crystal field.

In this work, we studied the nuclear magnetic polarization in single crystals of \( \text{NdFeO}_3 \) [1] and \( \text{NdAlO}_3 \) [2] by means of neutron diffraction experiments. Each crystal was rigidly attached at the desired orientation to the mixing chamber of a \(^3\text{He}/^4\text{He}\) dilution cryostat. The single degree of freedom was the rotation of the full set-up around the vertical axis, which provided access to one plane of the reciprocal space. Additionally, we studied powder diffraction patterns of \( \text{NdScO}_3\), \( \text{NdInO}_3\), \( \text{NdCoO}_3\) and \( \text{NdGaO}_3\). The enhancement of the magnetic reflections below 500 mK was consistent with the polarization of nuclear spins by a hyperfine field \( B_{hf} \) proportional to the electronic moment \( \mu \), \( B_{hf} = K \mu \), with \( K = 108 \, \text{T}/\mu_B \), very close to the theoretical calculation that gives \( K = 113 \, \text{T}/\mu_B \) [2].

As future prospects, the hyperfine polarization of other atoms can be studied, using a new especially designed set-up, which could overcome the difficulties and restrictions of the experiments reported here.

Acknowledgments:
We acknowledge the facilities supported by the Hahn-Meitner Institut and Institut Laue-Langevin. Also to the old Spanish Ministry of Education and Science, projects MAT02/166 and MAT2001-3507

References:
In-situ High Pressure Techniques for Small Angle Scattering Studies of Porous Materials

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In situ evolution of the nanopore structure of lamellar porous materials has been studied using small angle neutron and X-ray scattering (SANS and SAXS) under uniaxial stress. The mesoporosity (1 to 300 nm) was characterized along the parallel and perpendicular direction of the applied force using two kinds of high pressure cells. A specific cell \cite{1} that adapts to SANS studies of solid materials was developed in our laboratory at LPMCN. In this configuration the uniaxial load (up to 150 MPa) was applied on a flexible graphite sample via a piston connected to a hydraulic pump. The 2 mm thick sample was maintained between two monocrystalline sapphire windows allowing the neutron beam to cross the sample perpendicular to the stress. SAXS experiments were performed up to 300 MPa and 473 K using a low-pressure diamond anvil cell \cite{2} with the beam parallel to the direction of the stress. The combination of the SANS and SAXS techniques in these two configurations allowed the 3D description of the anisotropic porosity (figure 1) of an expanded vermiculite-based material during the compression and decompression processes. The concept of fractal geometry is used to characterize the surface irregularities between the pores and the matrix. To our knowledge this is the first in situ small-angle scattering study of porosity in a solid under pressure.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure1.png}
\caption{Small angle scattering patterns of expanded vermiculite compressed a) parallel and b) perpendicular to the incoming beam. c) The corresponding spheroidal shape of pores with the stress applied along the b axis direction.}
\end{figure}

\textbf{Acknowledgments:}
This research was supported by the Agence Nationale de la Recherche (ANR) through Materpro project ANR-08-MAPR-0011. We would like to thank A. Brület, F. Meneau, P. Lindner and J-P. Itié for technical assistance.

\textbf{References:}
\begin{itemize}
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In recent years the number of experiments where neutron scattering is used to study Soft Matter and Biomaterials is rapidly growing. The high complexity and variability of Soft Matter samples apply a set of special requirements on the design and operation of sample environment equipment. Here we are attempting to review existing Soft Matter and Biomaterials Sample Environment Zoo in a systematic way and look at perspective development directions. We are also discussing criteria for definition of Soft Matter and Biomaterials sample environment.
Catalysis in the Neutron Beam – Novel Sample Environment for in situ Experimentation at Industrial Conditions

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The characterization of catalyst is usually performed in the pressure range from UHV to ambient pressures, which is given by the used method. While nowadays catalyst are considered as dynamic materials whose active centres can be formed under reaction conditions, the extrapolation of catalyst properties over several orders of magnitude in pressure is often questionable.

Performing catalytic reactions under realistic pressures, e.g. 9 MPa and 723 K in highly reducing gases e.g. hydrogen/deuterium or carbon monoxide creates problems in materials choice concerning phase stability, resistance against hydrogen-embrittlement, catalytic-inertness, low neutron-activation, a high tensile strength, as well as good neutron transmissivity.

In the present contribution the construction and application of different continuous flow cells are presented, from which neutron diffraction data could be obtained during catalytic reaction at industrial conditions. By coupling online gas detection system, parallel structure and activity investigations are possible.

The successful application of the flow cells in two prototype reactions of heterogeneous catalysis, namely methanol synthesis over ternary Cu-catalysts at 6 MPa and 523 K [1] and ammonia synthesis over Fe-catalysts at 8 MPa and 700 K [2] is demonstrated.

Acknowledgments:
The authors would like to thank DFG (German research foundation, BE 4767/1-1) and Süd-Chemie AG for financial support. Friedrich Karl Seitz and Frank Rosowski (BASF) are acknowledged for providing the catalyst and fruitful discussions.

References:
Neutron Beam Fundamentals Development and Novel and Extreme Conditions at JRR-3 and J-PARC/MLF

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The developments of polarized neutron techniques, neutron focusing and detecting devices at JRR-3 and J-PARC/MLF in Tokai will be presented. The utilization of these techniques for neutron scattering experiments with extreme sample conditions will be reported. Special emphasis is put on the high-pressure sample conditions being developed both for JRR-3 \cite{1} and J-PARC/MLF \cite{2} together with the planned development of the focusing devices. Recent development on dynamical polarization experiment on polarizing SANS-J instrument \cite{3} will be also reported.

Acknowledgments: The developments of polarized neutron techniques, neutron focusing and detecting devices are in part supported by the Quantum Beam Fundamentals Development Program of MEXT, Japan.

References:
Recent Extreme Sample Environment Experiments at the ISIS Second Target Station

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The new high resolution magnetic diffractometer, WISH, fully exploits the opportunity offered by the high flux of cold neutrons available on TS-2 as shown by recent publications on small moment systems or small samples[1]. Right from the first drawing, a state of the art high field magnet was envisaged a central element of the beamline. I will describe a few key components of the WISH beamline that allow one of the latest additions to the ISIS sample environment suite, a recondensing 14T superconducting cryomagnet, to produce high quality data in high magnetic fields. To illustrate this, recent data taken on a powder and single crystal samples in this magnet down to dilution fridge temperatures will be presented. Measurements at high pressures [2] will be also be presented as well as recent experiments combining magnetic fields and pressure or electric fields.

Acknowledgments:
It is a pleasure to acknowledge the ISIS sample environment group.

References:
Novel and Extreme Sample Environment at the Helmholtz-Zentrum Berlin

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For a successful operation of a neutron or x-ray facility it is of particular importance to provide the scientific users the best technical and scientific support in order to perform highest impact investigations. The quality of the available sample environment support is certainly a key aspect for excellent scientific results. Sample environment at the Helmholtz-Zentrum Berlin is traditionally focused on extreme physical parameters like low temperatures or high magnetic fields, combined with a strong user support. Strengthening this expertise, the HZB is presently undertaking the project to build a high field magnet for neutron scattering. But in addition, two growing trends in neutron scattering are leading to the situation that the focus of sample environment is more and more shifted away from just only providing standard or extreme parameters. First, the increasing complexity of some neutron experiments requires the combination of neutron scattering techniques with complementary in-situ experiments or with in-situ sample preparation. Second, new user communities, especially in the area of soft matter, biology and solid state chemistry, push the development of novel specialized sample environment which is far from being standardized. In both cases sample environment is becoming a more important and more complex part of the neutron experiment itself. Sample environment at the HZB does already support these new developments and will further strengthen its efforts on these areas of research. In this presentation we will show our recent developments for extreme sample environments, in-situ experiments and novel specialized sample environment for soft matter, biology and solid state chemistry.

References:
Perspectives for Extreme Environment Indirect Geometry Spectroscopy at the European Spallation Source


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The European Spallation Source is a next generation neutron source for Europe, and at present across Europe instrument concepts are being developed for the ESS. In this presentation we wish to discuss a Swiss, Danish instrument concept for an extreme environment spectrometer at the ESS.

CAMEA (continuous angle multiple energy analysis) will be a spectrometer designed for optimal efficiency in the horizontal scattering plane. This is achieved by inverse geometry time-off-flight working in similar geometry to the Flatcone secondary spectrometer concept[1], but with essentially continuous angular coverage and multiple successive analyser crystals recording several energies per angle. As such it will be particularly well suited in combination with extreme sample environments, which limit neutron access to a plane. The high efficiency will also make it suited for parametric studies as function of static or stroboscopic environmental parameters likewise will the use of focusing optics and analyzers make it suited for small samples.

Combining this concept with the high flux expected at ESS will enable new kinds of studies. We shall present the design, projected performance parameters and examples of the scientific opportunities that will be enabled, with the aim to inspire considerations in and beyond the current community about the broader range of scientific opportunities CAMEA will enable. The result will feed into the scientific case and into the optimization of design parameters.

References:
Molecular Spectroscopy and Gas Handling Experiments in a Neutron Beam

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Molecular spectroscopy is a very powerful tool to study the dynamical properties of solid, liquid and gases. Inelastic Neutron scattering is a very powerful tool to study hydrogen-containing materials. With the development of neutron spallation sources, and the use of epithermal neutrons, inelastic neutron scattering can measure the vibrational spectra of materials on the whole range of vibrational motions (0-4400 cm$^{-1}$) and effectively opening up the field of neutron spectroscopy [1]. The recently commissioned Lagrange instrument at the ILL, although based at a reactor source can also access up to similar energy transfers. These sources have increased neutron fluxes and are making possible to increase the number of neutron studies of gas adsorption, catalysis etc. In this paper I will talk about the experimental challenges that appear when doing gas-handling experiments. In particular, cell designs, gas panels, centrestick designs, and present some experimental results with a critical view to the limitations and possible ways around them.

I will also present a new high pressure, high temperature cell designed to go up to 7 kbar of pressure of hydrogen and with a temperature range up to 230 C.

High pressure hydrogen cell, left design, right actual cell.

References:
The study of the microscopic mechanism governing water diffusion under high pressure is crucial for a variety of scientific issues spanning most of natural sciences. As an example, water diffusion in the minerals of the Earth’s mantle have strong incidence on the processes governing volcanic eruptions and intermediate-depth seismicity. Furthermore, water diffusion at depths below the reach of satellite observations is fundamental for oceanography. As last example, knowing in details the microscopic dynamics of this fundamental liquid under extreme conditions is essential in order to interpret observations and develop models of planet interiors [1].

On the other hand, water and other simple hydrogen-based liquids have always been key systems in the development of modern condensed-matter physics, because of their simple electronic structure and the peculiar properties deriving from the hydrogen-bond network. Their high compressibility and chemical reactivity have made these systems very challenging to study experimentally under static high P-T conditions. In the last few years, a large effort has been undertaken by several groups around the world [2] to extend the static and dynamic techniques to high temperatures and pressures, a program in which our group has been actively involved [3-5]. However, while the water structure is now well known up to 30 GPa, its dynamics was known only up to modest pressures (0.5 GPa) [6]. We have recently developed a new large-volume gasket-anvil ensemble for the Paris-Edinburgh press based on a toroidal design [7], which allows to perform quasi elastic neutron scattering measurements on hydrogen based liquids up to one order of magnitude higher pressures (5 GPa) respect to what was achievable with standard methods. The large volume HP press can be warmed up to 600K and the peculiar geometry of the gasket allows minimizing multiple scattering effects. This new device has been already settled up on IN6, at ILL, and on Focus at PSI, successfully showing that very high quality data can be obtained on liquid water, and more generally on hydrogenated liquids dynamics in such extreme conditions of pressure. The future implementation of the HP-QENS device to reach 20GPa and 1000K conditions will be also discussed.

Acknowledgments:
The ANR JCJC program is kindly acknowledged for the funding of the HPQENS project. ILL and SINQ are kindly acknowledged for highly valuable technical support and for beam time access.

References:
[2] Some of the most active groups in this field are the Geophysical Laboratory (USA) Lawrence Livermore National Laboratory (USA), CEA/DAM (France) and the Bayerisches Geoinstitut (Allemagne).
Concrete is widely used as a neutron shield and in building construction such as nuclear power stations, particle accelerators and medical hospitals. Concrete is very attractive for neutron shielding, because of concrete is contain of some elements (hydrogen, iron etc.) to moderating fast neutrons which are very penetrative. Boron is increased the neutron shielding effectiveness of concrete, since the boron isotope \(^{10}\text{B}\) has a high capture cross-section for thermal neutrons (over 3800 barns). Boron can be added to concrete different ways such as addition of boron to the water used in concrete or addition of boron containing natural minerals.

Neutron shielding capabilities of the sample can be described by total neutron macroscopic cross-section (\(\Sigma\)). It is the sum of the cross-sections for all the neutron-interaction processes such as the elastic and inelastic scattering reactions and neutron capture reactions ((n,\(\alpha\)),(n,\(\gamma\))).

In this study, the effects of addition boron and colemanite on the total macroscopic cross section of Portland concrete were investigated. Colemanite is one of the most important boron minerals and Turkey has the largest colemanite reserves in the world. Its closed formula is \(\text{Ca}_2\text{B}_6\text{O}_{11}\cdot5\text{(H}_2\text{O)}\). Also, colemanite can be used for shielding fast and thermal neutrons, since it is include both of hydrate and boron.

\(^{241}\text{Am-Be}\) neutron source with 74 GBq activity were used in our experiments. Average neutron energy of this source is approximately 4.5 MeV. BF\(_3\) detector with diameter 2.54 cm and length of 28 cm was used for counting neutrons. Also, Monte Carlo simulations were done for comparison of macroscopic cross section experimental results. Besides total macroscopic cross sections, absorbed doses and deposited energies by low energy neutron interactions were calculated using MCNP4C2 Monte Carlo code. The results have been compared with the standard shielding material of paraffin. Also, half-value layer (HVL) and tenth-value layer (TVL) were calculated and compared.

**KEYWORDS:** Neutron Shielding, MCNP Monte Carlo Code, Total Macroscopic Cross Section, HVL, TVL
A ‘portable’ 17 Tesla Cryomagnet for Small Angle Scattering

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The Birmingham 17T cryomagnet, specifically designed for beamline use, is a recently developed addition to the sample environment available to the neutron community and beyond [1]. It provides the largest currently available horizontal fields for small angle neutron scattering (SANS), and has also been used with X-rays. It can be transported to facilities in Europe, and has so far completed several journeys between the ILL, PSI, DESY, and its home base in Birmingham.

The solenoid design permits very high fields at a reasonable cost, trading this against angular access perpendicular to the field direction. An in-vacuo sample changing mechanism (Fig. 1) allows samples to be rapidly switched while the magnet is cold.

Samples can either be held in vacuum with rapid temperature control from 1.6 K to 300 K, or in ambient conditions using a room temperature bore insert.

We have recently been awarded an EPSRC grant to develop dilution refrigeration capabilities which will extend the temperature range down by a factor of 40.

Figure 1 The external vacuum chamber attached for rapid in situ sample change on the Birmingham 17 T cryomagnet. The picture shows the sample cup being locked into place at the centre of the magnet bore. The cup is held on the end of a rod passing through the cryomagnet windows, with all of the various tools required visible in front of that. (Photo: S.M. Hayden)

References:
The last ten years have seen a growing interest for the combined use of pulsed high magnetic fields and synchrotron or neutron techniques. However, high magnetic field experiments at large user facilities require a high duty-cycle. Within the framework of the MAGFINS project, the High Magnetic Field National Laboratory of Toulouse (LNCMI-T) has studied, optimized and designed a new coil with large angle conical access and cooling channels to increase the duty-cycle. The magnet produce an horizontal field in a bi-conical geometry, ±15 and ±30° upstream and downstream of the sample, respectively, that should allow neutron beam access over a wide range of scattering angles. It should generate pulsed fields up to 40 T with a rise time of 22ms every ~5-7 min using a 1.15MJ transportable pulsed field power supply developed in our laboratory.

This coil is presently under construction and is planned to be tested before the summer. It will be implemented into a cryogenic environment and used for neutron diffraction experiments at the ILL. Mechanical design is performed in close collaboration with the ILL cryogenics service, responsible for the N₂ and ⁴He cryostats.

Parallel to this work, tests of a pulsed reinforced polyhelix prototype are currently in progress to gain one step further toward even longer pulses and higher duty cycles.

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XtremeD - A New Diffractometer for High Pressures and Magnetic Fields

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Neutron diffraction has unique capabilities for the research under extreme conditions, mainly in two large areas: crystallography/geosciences and magnetism/solid-state physics. The growing interest for these problems is attested by the quantity and quality of publications, by the number of experiments proposed at the different neutron sources and by the new instrumentation projects under development all around the world. Therefore, the project for the construction of a “exreme conditions Diffractometer (XtremeD)” at Institut Laue-Langevin for both single crystals and powders, operating at high pressures (> 30 GPa) and high magnetic fields (up to 15-17 Tesla), is being considered and will be presented here.[1]

The scientific areas in which the projected instrument can make significant contributions and the main technical characteristics of the project are discussed in this presentation. The main idea of XtremeD is to combine a large solid-angle detector with an optional highly-focused beam on the sample, thus providing high flux while maintaining low background. The principal features of XtremeD will be the following:

– Optimisation for small samples: optional and variable focusing optics (choice of the focusing point).
– Large 2D position-sensitive detector for powders and single crystals.
– Sample environment adapted for high pressure (variety of pressure cells, up to 30 GPa for the moment, but with a goal of 50 GPa after the envisaged developments) and high magnetic fields (up to 15 Tesla in continuous mode, with the possibility of higher pulsed fields).
– Radial oscillating collimator and neutron shielding to suppress background
– Choice of monochromators (Si, PG), takeoff angles (40º-120º), and wavelengths (0.9–4 Å).

Figure 1. Scheme of XtremeD at its projected location.

Acknowledgments:
The Scientific Advisory Committee and the Working Team of XtremeD are acknowledged for the preparation of the scientific case and for the realization of the technical project respectively.

References:
Neutron Powder Diffraction Investigation and High-field Magnetization Measurements in Ammonium Iron(III) bis (hydrogenphosphate)


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The deuterated form of ammonium iron(III) bis (hydrogenphosphate), ND₄Fe(DPO₄)₂, was investigated from neutron powder diffraction data with a wavelength $\lambda = 4.724$ Å and from high-field magnetization measurements [1-2]. The crystal structure has two Fe positions (Fe1 and Fe2) with different local symmetry. The material undergoes two successive magnetic phase transitions with the magnetic moments lying in the ac-plane: one at $T_C = 17.82(5)$ K is related to the ferrimagnetic ordering of the Fe1 and Fe2 magnetic moments, $\mu_{FI} = 4.19(2)$ $\mu_B$ at 4 K; and the other transition is found to be at $T_t = 3.52(5)$ K due to an antiferromagnetic arrangement, with an equal moment antiphase structure, which is characterized by a long-period propagation vector close to $k_{AF} = (1/16, 0, 1/16)$ and a magnetic moment of the Fe ions of $\mu_{AF} = 4.41(3)$ $\mu_B$ at 1.5 K. High-field magnetization measurements up to 600 kOe show two multi-step metamagnetic processes. At low fields ($\sim 2$ kOe) a transition from the AF to FI phase occurs. The system remains in this state until reaching a critical field of around 180 kOe. At higher fields the Fe1 and Fe2 magnetic moments tilt away from the local axis, maintaining the total magnetization along the direction of the applied magnetic field, as the magnitude of the magnetic field steadily increases. The full induced ferromagnetic ordering is reached around 540 kOe. The low symmetry of its triclinic crystal structure and the complex pattern of competing superexchange pathways seem to be responsible for the existence of this double magnetic phase transition.

Acknowledgments:
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References:
POSTER PRESENTATIONS
Temperature and Pressure Dependent Single Crystal Neutron Diffraction of Fe(pmd)₂[Ag(CN)₂]₂ (pmd=pyrimidine)

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The combination of two or more properties in the same material to investigate possible interplay and synergy between them has been a singular topic of the scientific investigations nowadays. To move towards applications, one of the most promising fields is that of the switchable magnetic materials, which under different external perturbations, like temperature, light, pressure, electrical and magnetic fields, undergo drastic changes of their physical properties. Their molecular bistability can be therefore used to switch a second physical property. Spin crossover (SCO) compounds are particularly well adapted to this strategy since the spin state can be switched in a controlled manner between a low spin (LS) and high spin (HS) state involving magnetic, optical and structural changes stimulated by the action of temperature, pressure or light. Although much work has been done in this field, the subject of pressure dependent crystal structure determinations in SCO compounds is practically unexplored.

Here we focus in the structure of Fe(pmd)₂[Ag(CN)₂]₂ (pmd=pyrimidine) complex, that can be depicted as (4,4)-layers, where each Fe(II) atom is linked to four [Ag(CN)₂] anions which run parallel to the ac-plane. These planes are connected through the pmd ligands to the adjacent ones, constructing the final 3D structure. This compound is very sensitive to temperature and pressure (see figure 1), and undergoes a cooperative high-spin ⇔ low-spin effect.

Neutron diffraction studies with low temperature and high pressure have been performed at the VIVALDI instrument at ILL in order to obtain the crystal structure of the different pressure and temperature induced phases of the sample. The present work contributes to enlarge the short series of examples of structural studies under high-pressure reported up to now in SCO compounds.

Figure 1. Scheme of the structural changes undergone by 1 at 210K [left]. χ_MT vs T plot for 1 at several pressures showing the displacement of the spin transition over 300K. [right]

References:
Cryogen Free Low Temperature Sample Environment for Neutron Scattering Experiments

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Recent increase in liquid helium cost caused by global helium supply problems rose significant concern about affordability of conventional cryogenic equipment. The ISIS facility carries on an internal development program intended to substitute gradually all conventional cryogenic systems with cryogen free systems preferably based on pulse tube refrigerators. A unique feature of this cryo-cooler is the absence of cold moving parts. This considerably reduces vibrations and increases the reliability of the cold head. The program includes few development projects which are aiming to deliver range of cryogen free equipment including top-loading cryostat, re-condensing superconducting magnets and dilution refrigerators. Here we are going to describe the design of these systems and discuss the results of prototypes testing.
A Low Cost Cryogenic Goniometer

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A recurring problem with sample environment is the tilting of samples in the neutron beam. This is very difficult at base temperature as normal mechanical systems do not work well. This initiative was to see if a mechanically minimal system could provide variable sample positioning in two axes of rotation at base temperature. The system operates by moving part of a sphere against a ring support with three wires. The sample is positioned at the centre of the sphere on a sample mount. The complex operation of the three wires is controlled by an external computer system. The results have shown positioning to 0.1 degrees is possible without feedback.
The sample environment equipment suite necessary to tackle novel scientific challenges is more and more complex, diversified and requires a rigorous organization with well-known calibration curves, PID parameters, limits, communication protocols, algorithms, etc. In parallel, in order to reduce the investment and operation costs, these numerous devices must be shared by the neutron scattering instruments while remaining easy to install and remotely controllable as a single unit or in combination with others.

To cope with these difficulties, we propose to establish new standards and define a communication protocol that makes transparent the communication between the equipment and the electronics, analogous to the USB between peripherals and computers. This protocol, which would communicate on standard buses like CAN, RS-232, IEEE-488, IEEE 802.3, etc. would simplify a lot the installation of the devices and the diversification of the environments.

Today, the devices are sold with their own electronics and specific control software that often cannot easily be integrated in the instrument control software. We propose to develop with colleagues from other facilities and our main suppliers a USI standard that would reduce the efforts required to implement new sample environment equipment. A USI certified equipment would contain the information necessary to reset a list of electronics and implement algorithm in the sample environment control software. For example, it would suffice to plug a cryostat to reset the temperature and cold-valve controllers, the level monitors and implement the algorithm allowing to automatically regulate the sample temperature. Plugging a cryomagnet would reset the same electronics and activate other algorithms.

We present a draft of the USI protocol and its implementation at ILL. We also show how it can be integrated in a unified control software featuring dynamic management of the instrument configurations, a single graphical user-friendly interface, automatic logging of the events and safe remote control.
Sourcing, Assembly and Commissioning of 10 kbar H\textsubscript{2} Intensifier and Gas Handling System

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Hydrogen is seen as a clean and potentially plentiful energy source. The search for compounds that are capable of storing enough hydrogen and materials which could be used in efficient fuel cells is now an international priority. Neutron scattering is particularly suited for this purpose due to high sensitivity to hydrogen atoms. This is a vital tool to be able to probe materials and potentially promising for hydrogen technology under the extreme conditions such as high temperatures and high pressure. However these require sophisticated high pressure gas handling systems and intensifiers. Due to financial restraints the 10 kbar Hydrogen Intensifier is being assembled and tested at ISIS by the Pressure & Furnace section. The Intensifier components have been purchased. The system assembly and tests are in progress.

Notes:
World-leading Instruments Deserve World-class Environments

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The actual ILL sample environment equipment suite has been designed for instruments built a few decades ago. The experiments are slowed down by the performance of the equipment and the devices necessary to tackle novel scientific challenges are unavailable. We propose to establish new standards that will permit to exploit the performances of the new instruments and to develop or acquire the missing equipment.

In 2005, ILL has launched the ambitious project to modernize its equipment suite for controlling the environment in which an experiment can be carried out. These efforts have improved the reliability of our cryogenic equipment and increased the number and the quality of the experiments carried out at very high pressure or requiring sorption analysis.

But these efforts are insufficient: our team is still unable to satisfy the needs of the soft matter community and to provide the equipment that fits the performance of the new generation of instruments. Moreover, there is still some very ageing equipment and therefore a risk that ILL must reduce the number of high-pressure and very-low temperature experiments.

We therefore propose:

1) to provide a much better support to the soft matter community: Indeed, it is urgent to provide a much better support to the soft matter community with standardized state-of-the-art stop-flow observation heads, light-scattering setups, electric field cells, dedicated high-pressure cells, acoustic levitation chambers, rheometers and improved humidity chambers.

2) to secure and improve the high-pressure and very-low temperature experiments: We still use very ageing racks for controlling the pressure cells and dilution refrigerators. To secure and ease the use of these devices, we propose to build modern racks featuring fast and automatic change of pressure/temperature over wide ranges in cryostats and cryomagnets.

3) to improve the exploitation of world-class instruments with new magnets and high-performance equipment:

The IN5 ToF spectrometer and the D33 SANS instrument have been designed to host large cryomagnets that are presently unavailable at ILL. The highest horizontal field that can be applied on a TAS spectrometer is only 3.8 T. This prevents the neutron scattering community from tackling fundamental questions with high-performance instruments and we propose to acquire 3 new magnets, each with a dilution insert: a 10 T vertical-field magnet with a dedicated geometry for IN5, a 12 T horizontal-field magnet for D33 and a +7 T horizontal field magnet for TAS. Moreover, the cryostats, furnaces and dilution systems will be modified so that the times required to exchange or align the samples and to change the sample conditions will be strongly reduced.
Tof-CAMEA: An Extreme Environment Spectrometer Concept for ESS


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TOF-CAMEA is a new instrument concept considered for the European Spallation Source. The close to optimal coverage of the horizontal plane combined with good resolution and low background makes it ideal for an extreme environment spectrometer. To investigate this new promising design extensive simulations are performed and a prototype is under construction.

The analyzer-detector concept CAMEA (Continuous Angle Multi Energy Analysis) covers most of the horizontal plane with several analysers behind each other. They scatter different wavelengths out of the plane in a Flatcone like way but with its many analysers measuring several final neutron energies simultaneously for most in plane angles. Constructed as a reverse Time of Flight Spectrometer at the long pulsed source of ESS it will measure all incoming neutron energies in a broad interval simultaneously. Combined with the simultaneous measurements of a collection of variable outgoing energies at most in plane scattering angles this maps the horizontal part of \((q,\omega)\) space in a close to optimal way, while still allowing shielding and colimation to reduce background significantly.

The design is especially suited for an extreme environment spectrometer where magnets, pressure cells and other sample environments will often block the out of plane scattering. This will be further discussed by Paul Freeman. [1]

A prototype of the instrument is under construction and will be installed at the Paul Scherrer Institute to supplement the McStas Monte Carlo Simulations. The combined simulations and prototyping will allow proof of principle, test of critical parameters and provide realistic performance estimates.

References:

Nonmagnetic High Pressure Clamp Cells
For Neutron Scattering, Magnetic Measurements and NMR.

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High pressure began to be used actively in neutron scattering studies and magnetic measurements. For the TOF-method at the pulsed and steady state neutron sources, a number of high pressure clamp cells made from non-magnetic TiZr zero alloy, hard Al and hard HNU (NiCrAl) alloys are presented. All these cells are made for powder and single crystalline samples for diffraction and inelastic neutron scattering studies and can be put into standard cryostats (even in dilution fridge inserts) and high magnetic fields cryomagnets up to 6-10T.

For investigating the magnetic structure and the magnetization distribution in CePd(Rh):Si\textsubscript{2} under pressure at up to 40kbar and 10T with polarised neutrons on PRISMA (ISIS) \cite{1} and D3 (ILL) \cite{2} we have produced two new non-magnetic composite piston/cylinder type cells from TiZr+HNU. Single crystal or powder NaCl (for pressure calibration) and Fluorinert (pressure medium) \cite{4} were used in all experiments on the neutron sources SINQ (Swiss), ISIS (UK), HMI (Germany) and ILL (France). Some of these cells were used for the investigations of the magnetic spiral in ZnCr\textsubscript{2}S\textsubscript{4}, CsCuCl\textsubscript{3} \cite{5} and MnSi \cite{3} under pressure. The non-magnetic high pressure cell for magnetic remanence measurements up to 1.5 GPa was made from Ti (support) and hard HNU (NiCrAl) (insert) alloys \cite{6}. The results obtained with the nonmagnetic HPC up to 20kbar for NMR will also be presented.

References:
\begin{itemize}
  \item \cite{1} Quantum melting in magnetic metals, MJ Bull, SS Saxena, RA Sadukov, CD Frost, ISIS Faculty Annual Report 2001-2002, RAL-TR-2002-050, pp.48-49 (UK) http://www.isis.rl.ac.uk/isis2002/highlights/
  \item \cite{2} Magnetization distribution under pressure in the pressure induced superconductor CePd\textsubscript{2}Si\textsubscript{2}, N. Kernavanois, R. Sadykov, E. Ressouche, S. Raymond, P. Lejay and J. Flouquet, ILL Annual Report 2004/Highlights/
  \item \cite{3} Pressure dependence of the magnetic structure of the itinerant electron magnet MnSi, B. Fak, R.A. Sadykov, J. Flouquet, G. Lapertot, J. Phys.: Condens.Matter 17 (2005) 1635
  \item \cite{4} Hydrostatic limits of Fluorinert liquids used for neutron and transport studies at high pressure, V.A. Sidorov, R.A. Sadykov, J. Phys.: Condens.Matter 17 (2005) no.40,S3005
  \item \cite{5} Pressure Dependence of the Magnetic Spiral in CsCuCl\textsubscript{3}, Norbert Stuesser, Ravil Sadykov, Andreas Hoser, European Conference on Neutron Scattering, 25-29 June 2007, Lund, Sweden. Poster W174, 586
  \item \cite{6} Nonmagnetic high pressure cell for Magnetic remanence measurements up to 1.5 GPa in a superconducting quantum interference device Magnetometer, Ravil A. Sadykov, Natalia S. Bezaeva, Alexander I. Kharkovskiy, Pierre Rochette, Jerome Gattaceca and Vladimir I. Trukhin, Rev. Sci. Instrum. 79 (2008) 115102
\end{itemize}
General Information

Social Program

Welcome Cocktail
Pre-registered delegates and accompanying persons are kindly invited to the welcome cocktail to be held on the 27th of May 2012 at 20h30 at the
A - Hotel Melia Zaragoza
Cesar Augusto Avenue, 13
50.004 - Zaragoza
Spain

Lunch
Lunches as indicated in the daily time table will be held at:
B - Hotel Husa Zaragoza Royal
Arzobispo Domenech, 4
50006 Zaragoza
Phone: 976 21 46 00

Gala Dinner
The Gala Dinner is scheduled for May 30th at 20h30 at the
C - Restaurante “La Bastilla”
Calle del Coso, 177, 50001 Zaragoza
Phone: 976 29 84 49

All participants are kindly invited to attend.

Other useful Addresses:
Venue:
D - Edificio Paraninfo
Pza. Basilio Paraíso, 4
50001 Zaragoza