Inside nmi3

Issue 3 - May 2012

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- Schools for 2012
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SEVENTH FRAMEWORK PROGRAMME

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Editorial

The Scientific Advisory Committee (SAC)* of the first FP7 NMI3 programme (NMI3-I February 2009-January 2013) has become the Advisory Committee (AC)** for the new project (NMI3-II February 2012 – January 2016). What difference does this make? The ´S´ missing in the new acronym will result in extended tasks and more work for us to do! The members of the Committee, themselves, are responsible for this, since they are the ones who during the NMI3 General Assembly in Barcelona (May 2010) asked fundamental questions: what is expected from the Committee, and how do we achieve this objective?

Previously, the role of the SAC was mostly to evaluate the scientific output of the Joint Research Activities (JRAs). Today, the AC consists of six members, preferably independent from the partner institutions. As a group, they cover most of the topical and instrumental aspects of the Neutron and Muon techniques, and bring together the points of view of the user communities and the facilities staff. This Committee is therefore ideally placed to give an overall view on the progress made in the different activities of NMI3: not only the JRAs but also the Transnational Access and Educational programmes and Dissemination activities. For this purpose, it has been decided that the AC members would be invited as observers to all meetings and would be given all documentation relevant to the current projects. This will give the AC more opportunities to meet the people engaged in the different activities and to follow the progress made or the difficulties encountered, in a timely fashion.

*SAC- NMI3-I
- Götz Eckold (University of Göttingen, Germany, German Neutron Community)
- Kenneth Herwig (Oak Ridge National Laboratory, USA)
- Ted Forgan (University of Birmingham, UK)
- Françoise Leclercq-Hugeux (CNRS Lille, France, French Neutron Community)
- Romano Rinaldi (University of Perugia, Italy)

**AC- NMI3-II
- Götz Eckold (University of Göttingen, Germany, German Neutron Community)
- Kenneth Herwig (Oak Ridge National Laboratory, USA)
- Françoise Leclercq-Hugeux (CNRS Lille, France, French Neutron Community)
- Don Paul (Warwick University, UK)
- Romano Rinaldi (University of Perugia, Italy)
- Peter Schurtenberger (Lund University, Sweden)

The objectives are multiple:
- To ensure a better communication between the coordinators of the different activities and the AC members
- To enable the AC to participate valuably to a mid-term evaluation of the project and to discuss this analysis with the coordination team in order to detect rapidly if parts of the activities need to be reoriented
- To provide support for the evaluation made by the EC delegates
- To anticipate the preparation of next calls within HORIZON 2020 (FP8)
- To support further development of the European Neutron/Muon Research Area

It is worth emphasising that we can only provide advice to the NMI3 governing body. However, we expect feedback from the stakeholders, the activity coordinators and the management team on our suggestions.

The first evaluation from the AC will concern the European Neutron and Muon Schools supported by NMI3 during 2012.

As a conclusion, if I am allowed to quote Juliette Savin and Helmut Schober (editorial of the first issue of Inside NMI3): “Good communication lies at the heart of Innovation”, this ambitious task for the Advisory Committee will rely on mutual information and reactive feedback.

So please do not hesitate to contact us and to let us know what is going on!

Françoise Leclercq-Hugeux
Chair of the SAC for NMI3-I

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Focus on Joint Research Activities

In this issue of *Inside NMI3* we update you on the work carried out as part of the Sample Environment Joint Research Activity, started in February 2009 and ongoing until February 2013.

The Sample Environment JRA

The Sample Environment JRA is a far-reaching collaboration whose aim is to enhance the capabilities of sample environment available at European neutron facilities, in order to enable users to expand their exploitation of these facilities. New sample environments will open up new territories for hydrostatic pressure, temperature and advanced gas adsorption facilities; pushing at the boundaries of *in-situ* experimentation. Significant progress has been made in overcoming the technical challenges associated with these advances in sample environment. This has been greatly aided by the continual sharing of knowledge and experience between the technical groups at member facilities, FRM II, HZB, ILL, ISIS , LLB and PSI.

Aerodynamic and electrostatic prototype furnaces are being completed at FRM II and ILL as part of the JRA. An 8 kbar pressure cell has been manufactured at LLB and is currently in use at ISIS and HZB. Material and technical research is leading to novel cell design to solve the difficulties involved in producing a hydrogen-safe neutron-compatible high pressure cell. The array of equipment for gas adsorption measurements under extreme conditions that is being prepared at the HZB is astonishing. In addition, a collaborative spin-off has been the design of a standard sample stick, which will ease the transition of users from one facility to another. This JRA is on course to provide a breath-taking array of new sample environment equipment and experience in much desired but technically very challenging areas, opening up new realms of scientific exploration.

Figure 1:
8 kbar inert-gas pressure cell

High-Pressure Gas Cells

High-pressure research is one of the fastest-growing areas of natural science, and one that attracts such diverse communities as those of physics, bio-physics, chemistry, materials science and earth sciences. In condensed matter physics there are a number of highly topical areas such as quantum criticality, pressure-induced superconductivity or non-Fermi liquid behaviour, where pressure is a fundamental parameter. An increase in the range of available pressures up to 10 kbar for gas-loaded cells will make a significant impact on the range of science possible at neutron facilities. However, high pressure gas sample cells require thick cell walls which may lead to an unacceptable neutron background and thus the choice of materials and geometries is critical to improving the quality of the data. Reliable, safe and user-friendly high pressure gas handling systems are also an essential part of the development. One aim of this JRA has been to complement the 100 kbar Paris-Edinburgh equipment with an improved suite of gas pressure cells accommodating much larger samples up to 10 kbar for inert gases and to 8 kbar for hydrogen.
One important aim over the last 18 months has been to complete the design and manufacture of a 8 kbar inert gas cell (see figure 1), and after a design plan review, to begin the manufacture of a 10 kbar

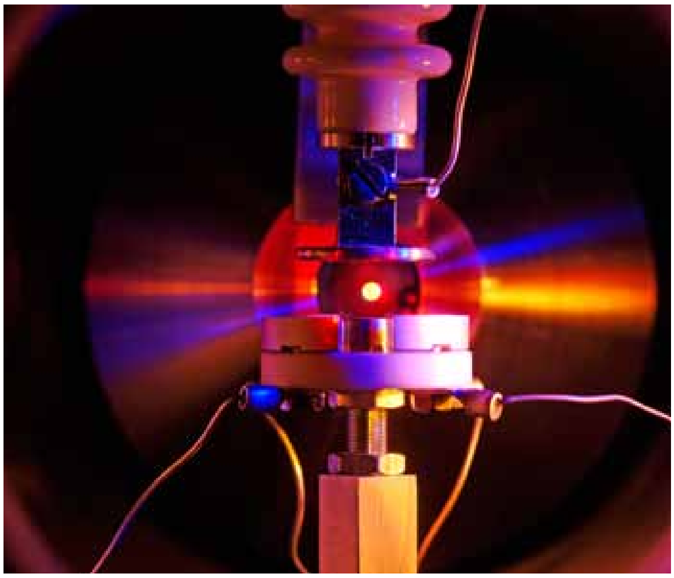
prototype inert gas cell. Hand in hand with the cell development will be the procurement of suitable 10 kbar automated gas handling systems for the LLB and STFC, in order to test and ultimately support the use of these cells on neutron beamlines.
Hydrogen is seen as a clean and potentially plentiful energy carrier. 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. Due to the high sensitivity to hydrogen, neutron scattering is particularly suitable for the investigation of promising materials for hydrogen technology, under extreme conditions such as high temperatures and pressures. However, these conditions require sophisticated gas handling systems and special sample cells. The development of cells for high pressure hydrogen is made difficult by the embrittlement of construction materials, and at present our knowledge of material behaviour in the presence of hydrogen is limited. Thus part of this JRA is focusing on developing suitable cell materials and technologies for the handling of hydrogen.

The design and manufacture of a 6 kbar hydrogen cell have progressed significantly during the last 18 months, and a design plan review for a prototype 8 kbar cell is almost complete and work has begun on the design. STFC is expected to have completed the procurement of an 8-10 kbar hydrogen handling system ready for testing the prototype cell.

Levitation melting

Studies of the liquid state are not only significant from a fundamental point of view but also represent important technological interests. The molten state is an essential stage in various industrial processes such as glass making, semi-conductor technology and the iron and steel making industry. However, the study of structure and dynamics in liquid metals or dielectric materials is often prevented by the chemical reaction of the high temperature melt with its sample holder.

The development of two types of levitation systems has been undertaken as part of this JRA, in order to offer greater access to high temperature ranges for neutron experiments involving a wider range of materials.



A sample in levitation. Picture courtesy of Prof. Andreas Meyer, Dr. Dirk Holland-Moritz Dr. Florian Kargl, Deutsches Zentrum für Luft- und Raumfahrt, Cologne.

The ultra-high temperature range can be achieved by the use of an electromagnetic (EML) or an electrostatic (ESL) levitation apparatus which allows container-less processing of samples. The design of the EML furnace for liquid metals was based on the prototype developed and built by the DLR in Cologne and adapted to dielectric materials by using an ESL system.
After completion of the drawings and design of the electrostatic levitation furnace, the furnace was manufactured and tested. A design review was completed and the furnace was then successfully tested on the TOFTOF neutron instrument at FRM II, during which the need for system modifications and the construction of a sample changer was highlighted. Following adjustments, the NESL (Neutron Electrostatic Levitation device) is now ready for use. Different experiments involving the levitation of conductive samples have been performed successfully at both FRM II and ILL.

Another aim of this task was to design and build a furnace using aerodynamic levitation for reaching temperatures up to 3000 K, combined with conductivity measurements. A novel levitator made form several nozzles has been designed and tested successfully. This technique applies the Bernouilli's principle and the Coandă effect so that the suction is sufficient to overcome the weight of the sample and the pressure differences tend to maintain the

Figure 3: Volumetrically controlled dosing of gases, vapours or mixtures, or alternatively operates in a continuous flow mode at HZB

sample on the axis. The design review is complete and the manufacture has started.
A video of a levitating sample in the furnace developed at ILL and FRM II, within the framework of the JRA, is available on the Sample Environment JRA page on the NMI3 website. [http://nmi3.eu/about-nmi3/joint-research-activities/sample-environment.html]

Gas Adsorption Control Systems

The technique of gas sorption is very important for the measurement of hydrogen storage materials and the characterisation of chemical and catalyst reactions in porous materials. The aim of this task is to significantly extend the range of systems available to allow real-time in-situ measurements of many diverse chemical and physical phenomena.
As part of this JRA aims, a volumetric low pressure (<1.5 bar) gas adsorption measurement system for experiments in an Orange cryofurnace (1.5-600 K) and, alternatively, in a cryogen-free miniature pulse tube refrigerator (50-600 K) have been developed. The development and tests of prototype *in-situ* gas adsorption temperature environments including sample stick and sample cells for the temperatures from 1.5 K to 600 K have been completed and tested in several neutron experiments.
These systems were enhanced by further developments to extend the temperature and pressure ranges up to 300 bar at 200°C. The system works in the specified parameters and allows volumetrically controlled dosing of gases, vapours or mixtures, or alternatively operates in a continuous flow mode

Figure 4: 200 bar injection stick commonly developed by HZB and ILL and adopted by ANSTO

(see figure 3). It may be operated remotely via a PC-controlled operation panel. For the general automation and control of different dosing processes a dedicated scripting language has been developed, but is still in the testing phase. In the future this software can also be used for the scripted operation of any complex sample environment hardware. The system will be released for user service after the first successful automated measurement of a vapour sorption isotherm.
The design of the high temperature extension up to 500°C for the magnetic suspension balance has been completed. The 100 bar pressure compartment has been constructed from a special Ti/Al-alloy with promising neutron properties, which needs to be tested in the beam. An optional thin copper insert is in construction to protect the system from embrittlement caused by use with hydrogen at extreme temperature and pressures conditions.
In addition, based on a prototype sample stick and gas sorption cells, the ILL, in cooperation with HZB and ANSTO, has developed an improved version of a gas sorption stick which combines volumetric, continuous flow and mixing applications, together with a standard series of sample cells designs (see figure 4).

Videos of the work carried out as part of the Joint Research Activity are available on the NMI3 website at http://nmi3.eu/about-nmi3/joint-research-activities/sample-environment.html

Activities

Highlights from our Access Programme

NMI3-II will continue to support access to neutron and muon facilities across Europe. In this issue of *Inside NMI3* we present to you highlights of research carried out thanks to our access programme.

Using muons to determine the role of Hydrogen impurity in oxides

By Rui Vilão

Transistors are key components of computer chips. Our ability to develop transistors of ever-decreasing size underpins the future of computing, if it is to follow Moore´s law for any longer. However, transistor miniaturisation is currently facing fundamental obstacles as these components reach atomic dimensions. One of the problems faced by transistor science is the thickness of the insulator layer of silicon dioxide SiO_2 used in the gate of transistors. In today´s transistors, this insulator layer is only nanometers thick. This means that it can be conductive even when very low tensions are applied and this can prevent the device from operating. The capacity of insulators to respond to an applied electric field can be quantified in a parameter called the dielectric constant. A possible solution to the problem of insulator thickness is the replacement of SiO_2 by more 'robust' insulators with a higher dielectric constant. Oxides with high dielectric constants are therefore currently under intense scrutiny. In this context, the investigation of the role hydrogen as an impurity has become increasingly important, especially since the discovery that this ubiquitous impurity may play a role in electron conductivity in zinc oxide ZnO. Theoretical schemes have also been proposed to explain the behaviour of hydrogen as an electrically active impurity or as an amphoteric impurity.

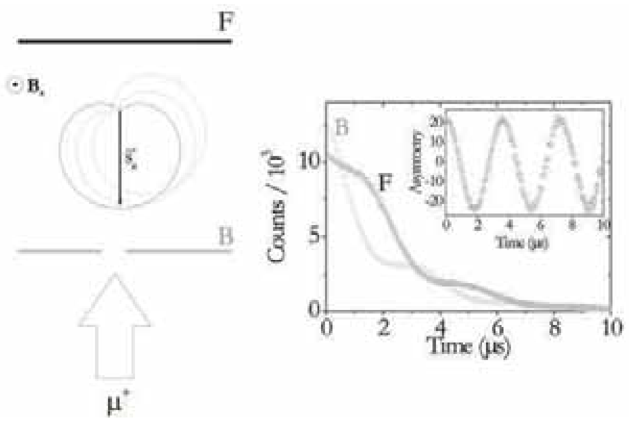


Figure 1: The basic muon spin rotation experiment. On the left, the basic setup: spin-polarised positive muons are implanted into a sample, in a region subject to a magnetic field Ba perpendicular to the initial muon spin, surrounded by two positron detectors in the forward (F) and backward (B) direction. The asymmetric probability for positron emission rotates with the muon spin and is drawn for three different times. The positrons counts NB and NF detected by detectors F and B form histograms like those shown in the right side. The experimental asymmetry A=(NB-NF)/(NB+NF) is shown as an inset.

We have recently addressed the characterisation of the isolated hydrogen configurations in the semiconducting oxide α- TeO_2 (paratellurite), a most interesting and relevant semiconducting oxide among nonlinear optical materials [1]. α- TeO_2 is a promising active material for optical devices and presents a relatively high dielectric constant. The presence of hydrogen in the fabrication process of this material leads to hydrogen incorporation in the lattice, and we were interested in investigating the effects of the incorporation of the isolated hydrogen impurity in the electrical properties of this material.

Using muonium to characterise hydrogen states

From the experimental point of view, the use of muonium, an atom composed of a positive muon as the nucleus [Mu≡μ+e-], as a light pseudo-isotope of hydrogen has become standard in order to obtain information about the electronic states of hydrogen in materials. The respective results compare well with those obtained with protons, for the very few cases allowing comparison. We have thus performed muon-spin research (μSR) experiments at the EMU instrument of the ISIS Facility, Rutherford Appleton Laboratory, United Kingdom, and at the DOLLY instrument of the Muon Spin Laboratory at the Paul Scherrer Institut, Switzerland. Our colleague Apostolos Marinopoulos in Coimbra has performed the corresponding first-principle *(ab initio)* calculations.

Muon spin reveals muonium dynamics

In the muon spin rotation measurements, a nearly 100% spin-polarised beam of positive muons, with their spin antiparallel to their momentum was directed at a TeO_2 single crystal while a magnetic field was applied perpendicularly to the muon spin, as illustrated in Figure 1. These experiments take advantage not only of the possibility of producing muon beams with full spin polarisation (all the muon spins pointing in the same initial direction) but also of the fact that the muon decay into a positron has the positron emitted preferentially in the direction of the muon spin. The follow-up of the evolution of the pattern of emitted positrons thus allows to have a direct link to the corresponding evolution of the muon spin and magnetic momentum, turning the muon into a precise magnetometer in condensed matter. In their thermalisation process, muons can capture an electron and form muonium. In this exotic atomic species, the muon spin interacts very strongly with the electron spin through the so-called hyperfine interaction, yielding typical frequency patterns.

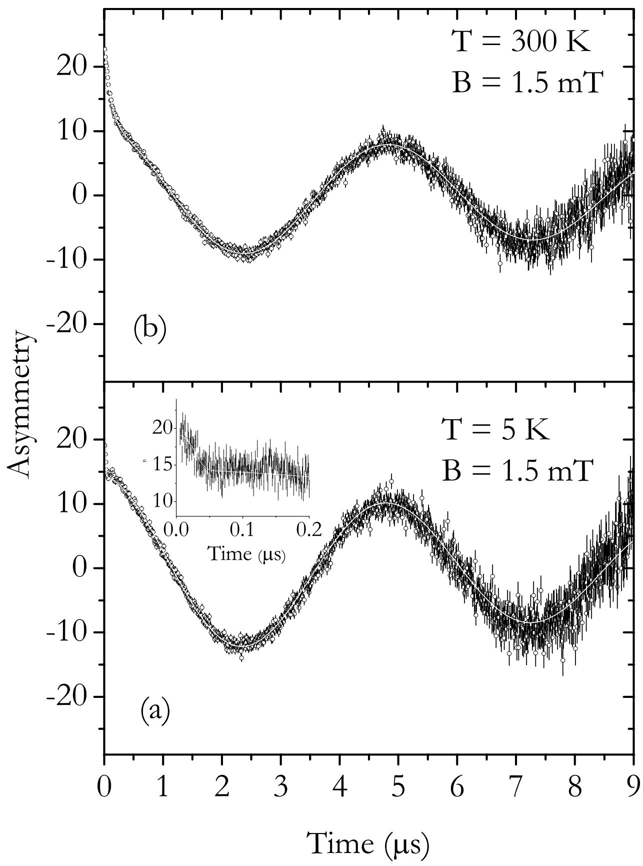


Figure 2: Muon spin asymmetry as a function of time, in transverse geometry (B = 1.5 mT), at T = 5 K (below), and at T = 300 K (observed at PSI). A clear nearly undamped oscillation at the Larmor frequency. A strongly relaxed signal is also observed in the first few hundred nanoseconds.

We could identify the muonium configurations involved through the corresponding frequency patterns. Further information about the muonium atom location and dynamics could be extracted from the relaxation of the muon spin polarisation, as well as from temperature dependence studies.

As shown in Figure 2, we observed two components with distinct spin relaxation rates, named fast and slow in the time spectra. The temperature dependence study revealed that a conversion process of the muonium states present is suggested by the fractions (Figure 3). We associated the low-temperature behaviour of the relaxation of the slow component (Figure 4) to the formation of a shallowly-bound muonium atom, with a hyperfine interaction of only about a few hundred kHz. In order to obtain a precise value for the hyperfine interaction A associated to the fast component identified at low temperatures, we performed an experiment with the applied field parallel to the initial muon spin polarisation. The dependence of the muon spin polarisation with the strength of the external applied field allowed us to determine the value A = 3.5 GHz (Figure 5).

The slow and fast fractions identified were assigned to a shallow-donor configuration at an oxygen-bound site and to a deep-acceptor configuration at an interstitial site, respectively. In the shallow donor configuration, an electron is suggested to be bound to the muon with an extremely small ionization energy of only about 6 meV, so that this centre effectively contributes with electrons to the conduction band and to n-type conductivity. In contrast, the deep acceptor configuration corresponds to an atomic-like muonium centre where the electron is tightly bound with and ionization energy of the order of the eV. The temperature dynamics is likely to correspond to delayed electron capture by the deep-acceptor muonium, forming the negatively charged state Mu^- [1]. This centre thus tends to behave as an acceptor, removing electrons and contributing to p-type conductivity.

H as an amphoteric impurity in TeO_2

The present experimental results, in association with the *ab initio* calculations, clearly identify the basic donor and acceptor configurations of isola-

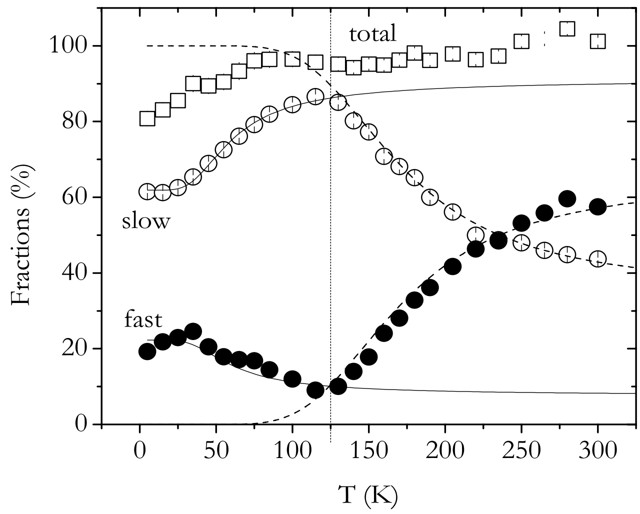


Figure 3. Temperature dependence of the slow component (open circles), the fast component (solid circles) and total fraction (open squares), as observed at PSI, for an applied transverse field B =1.5 mT. A clear (inter)conversion process occurs between these two components.

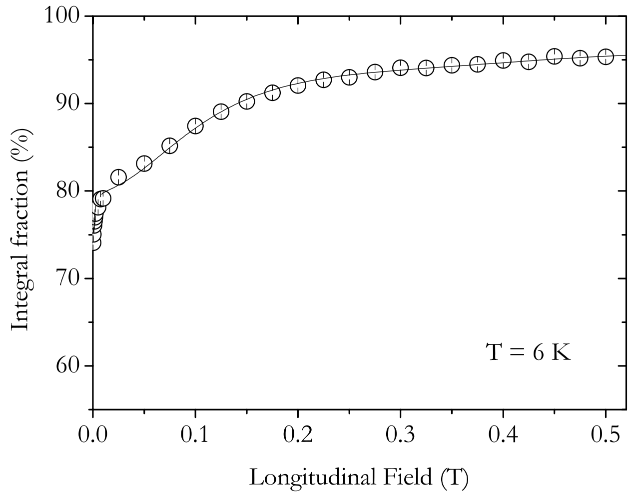


Figure 5. Dependence of the integral fraction with an applied longitudinal field, at T = 6 K, observed at ISIS.

ted hydrogen in TeO_2. Moreover, the corresponding levels are suggested to be inverted in the band gap, so that the binding energy of the second electron in the H- configuration is larger than the binding energy of the electron in the H0 configuration. As a consequence of this inversion of levels, if the material is electron-deficient (p-type), hydrogen will tend to donate its electron to the conduction band, acting as a donor. If the material is electron rich (n-type), hydrogen will tend to capture an extra electron from the conduction band, behaving as an acceptor. In short, hydrogen will tend to always counteract the prevailing conductivity, and to act as an amphoteric impurity.

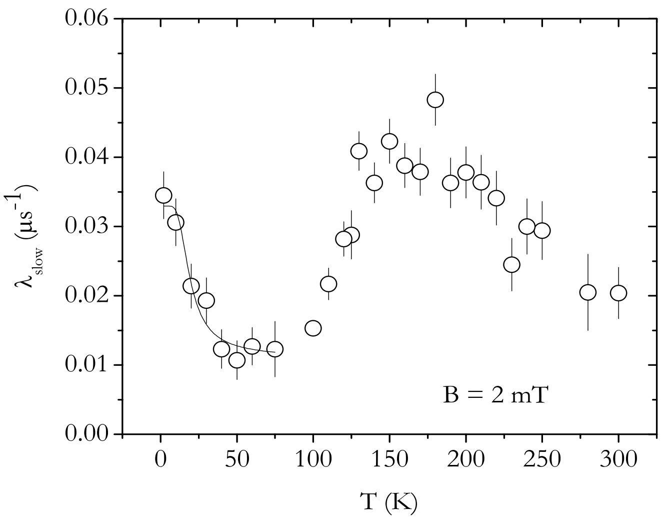


Figure 4: Temperature dependence of the relaxation of the slow component, observed at ISIS in transverse-field experiments (B =2 mT). The solid line below 75 K is a fit to an activated process which we assign to the ionization of a shallow donor. We associate the peak around T = 175 K to charge fluctuations arising from the (inter) conversion process.

Rui Vilão is Assistant Professor at the Department of Physics of the University of Coimbra. He is a member of the Group of electronic and magnetic properties of materials at the Centre for Materials Research by X-ray diffraction.

Reference:

[1] R. C. Vilão, A. G. Marinopoulos, R. B. L. Vieira, A. Weidinger, H. V. Alberto, J. Piroto Duarte, J. M. Gil, J. S. Lord and S. F. J. Cox, Hydrogen impurity in paratellurite α-TeO_2: Muon-spin rotation and ab initio studies, Phys. Rev. 84, 045201 (2011)

Muons reveal unconventional magnetism in a cupric-oxide analogue

By Andrej Zorko

Following the breakthrough discovery of high-temperature superconductivity, copper oxides have been the focus of intense research for almost three decades. The link between superconductivity and magnetism coexisting in some of these materials is still unexplained. In the basic, conventional copper oxide materials, the atomic spins all line up together at low temperatures - something known as long-range magnetic order. However, a fascinating array of other behaviours can also be found in copper oxides, depending on how the atoms are arranged. A particular focus has been on geometrically frustrated lattices, whose triangular geometry prevents simultaneous energy minimisation of all bonds of nearest-neighbour spins and thus leads to unconventional magnetic states [1]. The frustration is strong on spin lattices with a triangular motif when the dominant nearest-neighbour exchange interaction favours antiparallel alignment of interacting spins, as depicted in Figure 1.

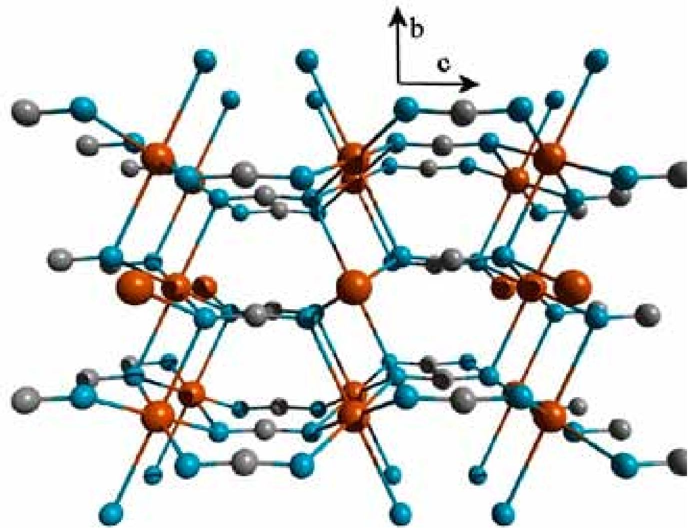


Figure 2. Crystal structure of CuNCN: orange, blue and grey spheres denote copper, nitrogen and carbon ions, respectively. According to Ref. 2 the appropriate spin model of the system is a 2D spatially anisotropic triangular lattice in the crystallographic ab plane, while Ref. 3 claims CuNCN to be a quasi-1D system with spin chains running along the c crystallographic axis.

Replacing oxygen with nitrogen environment

Copper carbodiimide, CuNCN, is a new copper-based antiferromagnet that has been recently crystallised and could potentially possess unconventional magnetic properties. Its NCN^{2-} groups are chemically equivalent to O^{2-}, yet much larger than these ions. Therefore, CuNCN is a chemical analogue of the cupric oxide, CuO, but its electronic properties might be very different. Indeed, from the magnetic point of view, CuNCN features magnetic susceptibility that is suppressed compared to copper oxides by one order of magnitude. In addition, it apparently lacks the conventional long-range order at least down to 2 K [2], in clear contrast to CuO that orders antiferromagnetically at 231 K. Replacing oxygen with other anions thus provides an exciting opportunity to extend the class of materials with exotic electronic properties and to eventually control these properties by chemically modifying the exchange bridges. Numerical investigations of CuNCN [2,3] agree that the reduced susceptibility should result from very strong exchange couplings, however, they disagree on the dimensionality of the appropriate spin model for CuNCN. The first of the proposed models features a frustrated triangular lattice [2], while the second one predicts a much less frustrated quasi-1D lattice [3]. An experimental verification of the presence/absence of the conventional order at low temperatures could resolve the issue of the appropriate model, since the latter model predicts magnetic ordering below around 100 K.

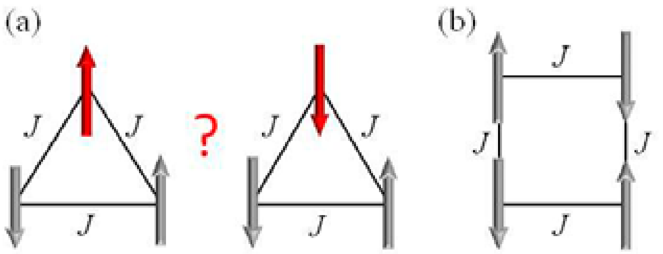


Figure 1. Geometrical frustration prevents simultaneous antiparallel alignment of spins on all antiferromagnetic bonds J (a) on a triangle, (b) as opposed to a square.

μSR insight to magnetism in CuNCN

In our attempt to determine the ground state of CuNCN [4] and settle on the appropriate spin model, we performed muon spin relaxation (μSR) measurements at the MUSR facility (ISIS, Rutherford Appleton Laboratory, United Kingdom) and at the GPS facility (Paul Scherrer Institute, Switzerland). This local-probe experimental technique is especially suited for cases where other experimental methods fail, because it is extremely sensitive and can detect very small local fields, through precession of muon polarisation. In addition, it can easily di-

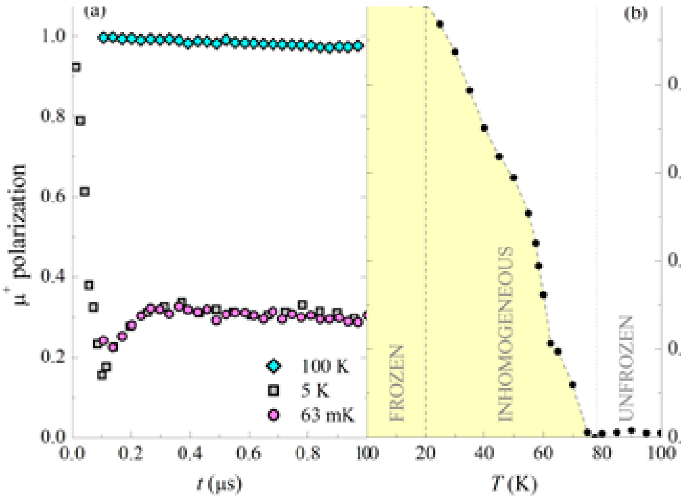


Figure 3. (a) Relaxation of μ+ polarisation in zero applied magnetic field. The shape of the relaxation curve changes below 80 K and develops a dip, characteristic of disordered frozen local magnetic fields. (b) Muon relaxation experiment in weak transverse magnetic field reveals a broad inhomogeneous region of coexisting dynamical and static local fields at muon sites, extending between 20 and 80 K.

stinguish between static and dynamical local magnetic fields. When fields are static and long-ranged, the projection of the muon polarisation on a given axis will coherently oscillate, while dynamical fields will lead to a monotonic decrease of the muon polarisation.

Random freezing in CuNCN

In order to probe the nature of the internal fields at muon stopping sites in CuNCN, we first performed μSR experiments in zero applied magnetic field. A clear change of the μSR relaxation curve around 80 K (Figure 3a) from a monotonic curve at high temperatures to a non-monotonic curve at low temperatures immediately reveals spin freezing that occurs around 80 K. However, in contrast to all numerical predictions, from the specific shape of the low-temperature depolarisation curves we can unambiguously conclude that the frozen-spin state lacks long-range order down to at least 63 mK (Figure 3a). Thus, the state rather resembles a spin-glass. We've further inspected the spin-freezing process by applying weak transverse magnetic field. In this μSR experimental setup, the initial muon polarisation is a direct measure of the fraction of muons sensing no static internal fields. In CuNCN the process of spin freezing is extremely slow, as it extends from 80 down to 20 K (Figure 3b).

Unconventional magnetism in CuNCN

The spin-glass-like behaviour observed in CuNCN is by no means conventional. CuNCN lacks usual fingerprints of the spin-glass transition typically observed in bulk magnetization or specific heat measurements [4]. Moreover, the spin freezing process is extremely slow, as we have observed an inhomogeneous phase with regions of both frozen and unfrozen fields coexisting between 80 and 20 K. The stability of this inhomogeneous phase in such a broad temperature region is unprecedented for canonical spin glasses. Therefore, we have explored another interpretation of the μSR data, in which the observed spin freezing would be muon-induced [4]. However, our recent nuclear magnetic resonance experiments speak against this scenario. The observed unusual magnetic state of CuNCN thus apparently stems from the significant geometrical frustration of the corresponding spin lattice, and thus speaks against the theoretically predicted 1D spin model.

Andrej Zorko is scientific collaborator at the Jožef Stefan Institute, Ljubljana, and at the EN–FIST Centre of Excellence, Ljubljana, Slovenia

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A new approach to cancer therapy through Ru/nucleolipid nanovectors

By Luigi Paduano

Ruthenium complexes could represent a new and promising route to a safer, more effective cancer therapy. The potential anticancer activity of this metal has led to increasing interest from the research community. Indeed, Ruthenium complexes are less toxic than Platinum-based compounds, which are currently successfully used for cancer treatment. Futhermore, ruthenium-based compounds are also selective towards metastases. Nevertheless, there are still questions regarding the effectiveness of Ruthenium complexes, mainly due to their poor stability in physiological conditions. In order to make treatment using Ruthenium more effective, we decided to develop potential molecular vectors, capable of transporting Ruthenium (Ru) in large quantity and preferentially to the tumor site, and to shield it against environmental degradation over long circulation times. With these goals in mind, we designed novel Ru-based amphiphilic molecules containing one or two long hydrophobic hydrocarbon tails, as well as one hydrophilic head present on the scaffold of the molecules (see Figure 1). These molecules readily self-assemble into ordered nano-sized aggregates, which were studied using Small Angle Neutron Scattering (SANS) measurements performed on the JCNS instrument SANS1 at FRM II.

Ru-based aggregates

The Ru-based molecules were synthesised using nucleosides made of uracil or thymidine and a sugar ring, as a scaffold. The sugar ring was used to attach one or two long aliphatic, hydrophobic chains, together with a hydrophilic polyethylene glycol (PEG) chain of variable length to act as a protective stealth agent for the metal complex nano-aggregates. Such molecular architecture ensures low toxicity, high biocompatibility and the capacity to penetrate the cell membrane, whose main components are phospholipids.

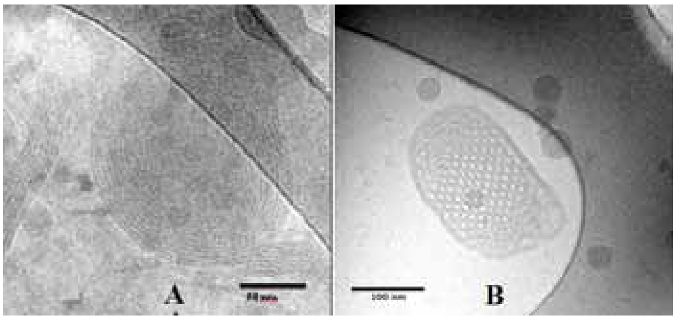

Figure 2. Cryo-TEM images of multilamellar vesicles (A) and cubosome-like particles (B) formed by some Ruthenium/POPC systems..

Information on the supramolecular organisation of the nano-aggregates was achieved through Small Angle Neutron Scattering (SANS) measurements. The SANS measurements showed the formation of layered vesicles (Figure 2A), whose compactness also depends on the length of the PEG chain, and cubosomes (Figure 2B). It is important to outline that the ideal size of the nano-aggregates to be used for drug delivery ranges between 50 and 1000 nm, and this condition was fulfilled by all the amphiphilic Ru-complexes synthesised.

Optimisation of chemotherapeutic effect

Two strategies were subsequently followed in order to optimise the chemotherapeutic effect of these compounds. One one hand, we inserted a choles-

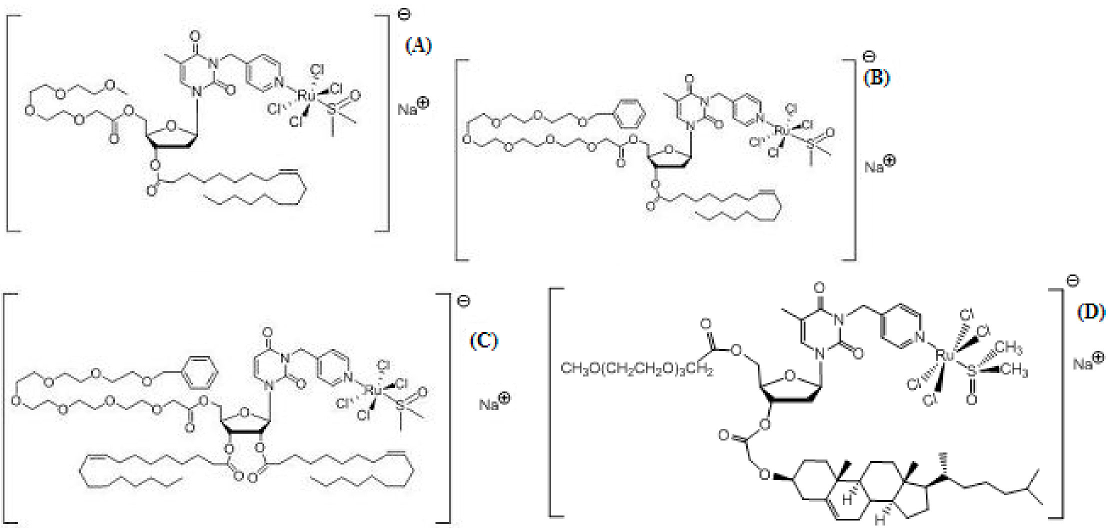

Figure 1. Synthesized nucleolipid-based Ru(III) complexes: ToThyRu (A), HoThyRu (B) and DoHuRu (C), ToThyCholRu (D).

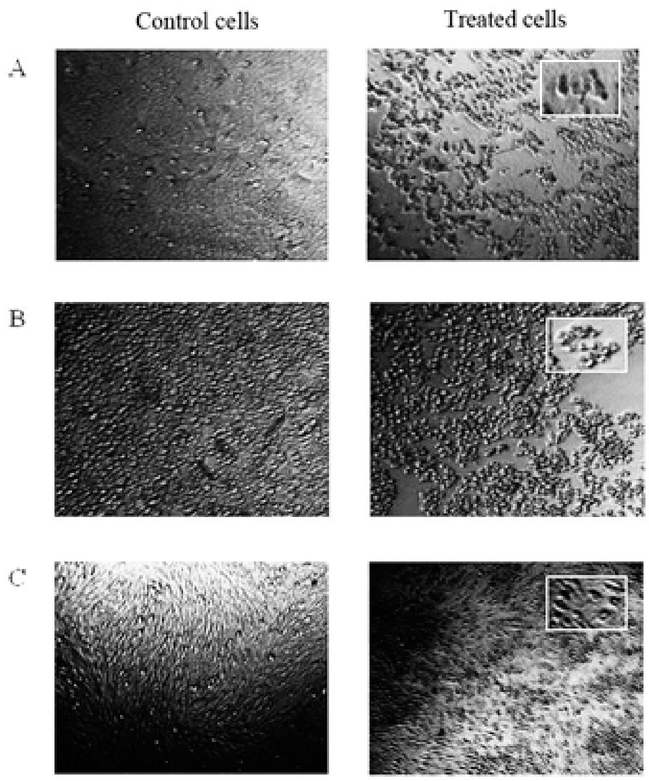

Figure 3. Microphotographs by phase-contrast light microscopy of two cell lines untreated (control cells, left column) or treated for 48 h with ToThyRu/POPC (right column). Inset: higher magnifications of injured cells following incubations with the antiproliferative ToThyRu/POPC liposome.

terol group on the thymidine scaffold to improve the Ru-complex delivery to cancer cells, and on the other, we incorporated the synthesized molecules into a protective liposome aggregate (POPC). The resulting nano-aggregates contained up to 15 per cent molar concentration of the Ruthenium complex and were stable for several weeks.
We tested these doped Ru-complexes in-vitro by adding them to live cultures of human cancer cells. Our tests showed that they are effective in inhibiting the growth of the cells (see Figure 3).

In summary, we are confident that the approach followed by incorporating Ruthenium complexes in biomimetic nano-systems offers enormous potential for chemotherapy (Figure 4). The small-angle neutron scattering technique represented an essential tool in characterising the morphology as well as the microstructural characteristic of these valuable therapeutic candidates.

Luigi Paduano is professor of physical chemistry at the Chemistry Department of University of Naples Federico II in Italy.

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[2] L. Simeone *et al.* A Cholesterol-based Nucleolipid-Ruthenium complex stabilized by lipid aggregates for antineoplastic therapy. Bioconjugate Chem (2012), article in press (DOI: 10.1021/bc200565v).
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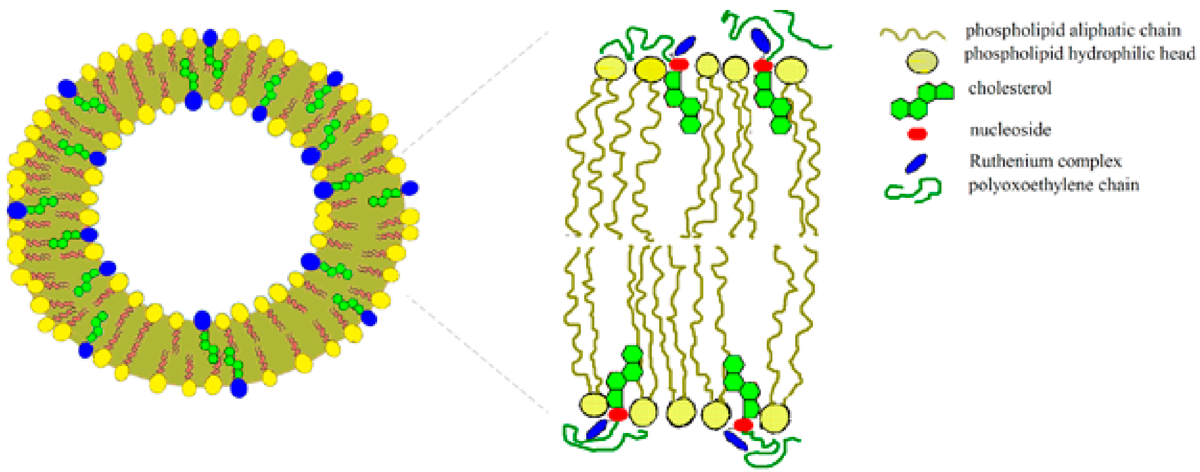


Figure 4. Schematic representation of Ruthenium/POPC vesicles.

News from the facilities

Joel Mesot reappointed Director of PSI

Professor Joël Mesot has been reappointed director of the Paul Scherrer Institute (PSI), for a further four years by the Swiss Federal Council. His second term will begin on August 1st 2012. Dr Mesot joined PSI in 2004 as Head of the Laboratory for Neutron Scattering. Before that he had spent a few years in France and the USA, after a doctorate in physics from the Swiss Federal institute of Technology Zurich (ETH Zurich). In this interview with Juliette Savin, Joël Mesot tells us a little bit about himself, and the future of neutron and muon research at PSI.

Inside NMI3: What was your reaction when you learnt that you were reappointed Director at PSI?

Joël Mesot: First of all, it felt good to know that the Swiss Federal Government appreciated the results PSI achieved during my first term of directorship and thus entrusted me with another term. And I am glad to be able to contribute to PSI's future in interesting times: At the end of my next term, the X-ray laser SwissFEL, PSI's new large-scale facility, will be commissioned. Furthermore, there are great challenges when it comes to future energy supplies, and we have to think how PSI, as Switzerland's largest energy research institute, can contribute best to the necessary solutions. So these will be challenging years, and I am looking forward very much to playing an active role in developments.

Inside NMI3: Could you tell us a little bit about the place of neutrons and muons in the future strategy of the institute – especially now that SwissFEL is underway and that Switzerland has joined the ESS collaboration?

Joël Mesot : It is part of PSI's mission to operate world-class large-scale research facilities for the Swiss and international research communities. And it is a characteristic of PSI to run an almost unique suite of facilities on one campus. The neutron and muon facilities, together with the Swiss Light Source, are, and will remain, essential parts of a suite that will become, once SwissFEL becomes operational, truly unique in the world during the coming decade. The ESS will not be a competitor to the neutron research performed at PSI. As a pulsed neutron source, it will complement PSI's continuous source SINQ, extending research opportunities with neutrons for Swiss scientists in research fields for which SINQ is not really designed. Neutron research at PSI, and research in Switzerland in general, will also benefit from the ESS in that Swiss scientists and engineers will, through their various abilities and experiences, help develop the target and build instruments for the ESS.

Inside NMI3: And how much space will there be for development at the neutron and muon facilities at PSI?

Joël Mesot : First of all, we will make sure that the research opportunities at SINQ and at the SμS comply with the highest standards, and thus remain attractive to our most demanding users. We are also about to complete several large development projects – the new three-axis-spectrometer EIGER at SINQ and the high magnetic field platform at the SμS. Also, the Ultracold Neutron Source, a facility for investigating the properties of the neutron itself, is now running and should reach its attempted neutron density this year. So there is also space for development beyond maintaining the status quo. In coming years, the focus will be on strengthening the synergies between the PSI energy and biology/chemistry departments and our large-scale facilities.

Inside NMI3: Do you miss doing research yourself? How much time do you dedicate to it?

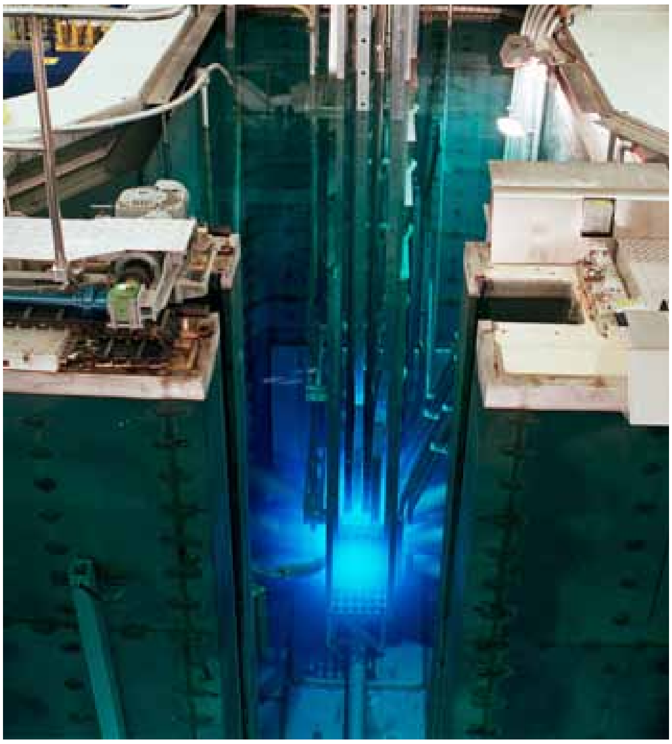
My goal is to devote one day per week to research. I am running a small research group, where I am working on magnetic materials and superconductors, and I am still supervising PhD students. Of course, I can do much less than I used to, and colleagues support me in the everyday work with the PhD students. But I think that this work is crucial for my task as Director, as it allows me to stay closely in touch with current scientific developments and not to lose sight of the realities of scientific work.


Picture: Scanderbeg Sauer Photography

New funding allows RID to start OYSTER programme

On the 20th January 2012, the Dutch Council of Ministers decided to give a substantial financial support to the Reactor Institute Delft. With this support the institute will implement the OYSTER programme (Optimised Yield – for Science, Technology and Education – of Radiation) and boost its capabilities as the centre of scientific expertise in the field of nuclear and radiation research and education in The Netherlands.


Picture: RID


Picture: RID

The aim of OYSTER is to increase the power of the research reactor, install a cold neutron source, improve the existing instruments and develop new innovative instruments, which will enable groundbreaking materials science research in the fields of health and energy. More specifically, a Positron Annihilation Lifetime Spectrometer will be built, employing the even more intense positron beam. In addition, a Cold Neutron Irradiation Facility, a Scanning Neutron Microscope and a world-class Neutron Diffractometer are now on the drawing boards.

The Reactor Institute Delft is the Dutch centre for multidisciplinary scientific research and education in reactor physics, neutron and positron radiation, radiation detection and radiochemistry. A large part of the research focuses on medical applications, such as the production of medical isotopes and the diagnosis and treatment of cancer. In addition, new materials are being developed for sustainable energy such as solar cells and batteries and work is being carried out to develop the nuclear reactors of the future. Furthermore, the Reactor Institute Delft is the organisation in the Netherlands that trains people to work with radiation. It also provides international workshops and training and is one of the thirteen worldwide Collaborating Centres of the International Atomic Energy Agency (IAEA).

The director of the Reactor Institute Delft and TU Delft dean, Tim van der Hagen said: “One of the things OYSTER will make possible is the development of a new generation of medical isotopes for detecting or destroying cancer cells. The slower neutrons will also help to drastically improve the efficiency of solar panels and the capacity, charge rate and safety of batteries.”

The Dutch government is also investing in the realisation of PALLAS, the new nuclear reactor for radioisotope production at Petten. In this way a very strong nuclear knowledge structure will be put in place, which will position the Netherlands amongst the global leaders in nuclear knowledge and product development.

The total neutron intensity of the modified reactor will be as a result of the OYSTER programme, the total useful neutron intensity of the RID reactor will be increased 100 times.

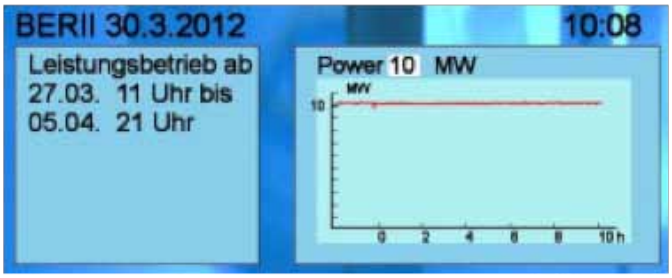
BER II back in operation

By Thomas Gutberlet, HZB

In October 2010, the BER II research reactor was shut down to prepare the upgrade for a new cold source and the exchange of an in-pile beam tube, after nearly 15 years of operation. Both efforts turned out to be time-consuming and complicated, but by joining forces, the work could be completed and the reactor was restarted on Tuesday 27th March 2012. The source reached is nominal thermal power on Thursday 29th March and the neutron instruments in the experimental and guide halls of BER II started to get back into normal user operation.

Parallel to this upgrade of the BER II reactor with the new cold source, which will increase the neutron flux by a factor of 1.6 for cold neutrons, all neutron guides in the neutron guide hall I have been exchanged and a number of instruments upgraded. The cold three-axis spectrometer FLEXX has been rebuilt and placed at an end position of the neutron guide. The tomography station CONRAD has been completely rebuilt and equipped with a new guide with larger cross section. The tof spectrometer NEAT will get a new guide and a new position with an extension of the neutron guide hall, which will be ready in 2014. A second tomography station PONTO is under construction and a test beam line for the ESS will be built in 2012.

Users are awaited to benefit from this upgrade and from the new instruments.



Advanced Seminar on Perspectives for Neutron Science in Novel & Extreme conditions

Next dates: 27th to 30th May 2012
Zaragoza, Spain

Frequency: irregular
This seminar is open to the wider scientific community and aims to identify the scientific drivers requiring novel and extreme conditions, as well as determine how neutron scattering techniques can contribute to this area of research. The seminar will bring together scientists, engineers and instrument specialists and aims to promote discussion and the exchange of ideas between these three groups. The organisers hope that this exchange will foster ideas for novel and extreme sample environment equipment and instruments, so that new scientific areas can benefit from neutron scattering techniques.

During the seminar, world-class experts will be invited to give tutorials on the following topics: high pressure, high magnetic and electric fields, soft matter equipment, low temperature and high temperature.

These tutorials will be followed by discussions on the technical aspects and future possibilities of the field being discussed. Current capabilities and limitations, but also the type of science that could be investigated provided that certain developments be made, will be addressed.
Registration to participate in the seminar is open until the 15th of April 2012. The event registration fee covers participation, a welcome reception, coffee breaks, lunch meals and a gala dinner.

For more information, please visit the website: www.unizar.es/m4/index.php/events

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NEUTRONS FOR SCIENCE

Picture of the ´Paraninfo´ of the University of Zaragoza (venue), courtesy of organisers.

Bombannes summer school

Next dates: 7th to 14th June 2012
Carcans Maubuisson, France

Frequency: every two years
The Bombannes summer school offers a practical approach to scattering methods, introducing today´s key techniques: neutrons, X-ray and light sources, to study structure and dynamics in systems containing colloids, polymers, surfactants and biological macromolecules.

The Bombannes school has been introducing the current methodology of static and dynamic scattering techniques and their application to soft matter systems, since the 1990´s. It provides advanced training to young researchers with a wowrking place in European laboratories at post-graduate and post-doctoral level.

The school consists in 25 hours of general lectures, divided into two parts. The first half of the week provides a general introduction to scattering experiments.
Basic concepts of data treatment, the notion of contrast, general theorems, instrumentation and resolution effects are introduced.

In the second half of the week, applications of static and dynamic scattering techniques to investigate typical soft matter systems such as colloidal suspensions, microemulsions, micelles and surfactant solutions, polymers, biological systems and turbid suspensions are presented. Evening sessions are dedicated to student presentations and contributions.

The number of participants is limited to 35.

For more information please visit the website: www.ill.eu/bombannes

Students and lecturers at Bombannes 2010: Picture courtesy of the organisers

Giornate Didattiche/SISN school

Next dates: 23rd June to 3rd July 2012
San Giovanni in Valle Aurina, Italy and
Grenoble, France

Frenquency: annual
The GD is the school on neutron scattering organised by the Societá Italiana di Scattering di Neutroni (SISN). The school is generalist and is aimed at university students, PhD students and young researchers from different disciplines. The school is delivered in Italian and it provides a very general introduction to the various neutron scattering techniques.

The school is divided in two parts: the first part provides a theoretical introduction to neutron scattering and the different neutron scattering techniques. The students also perform exercises on didactical examples in small groups supervised by a tutor. They are required to participate actively in these exercises. For the second part, the students are transferred to the Institut Laue-Langevin (Grenoble) and take part in experiments on the Italian Collaborative Research Group instruments BRISP and IN13 and on one diffractometer.

For more information please see the website: www.sisn.it/

Valle Aurina, Italy, where the first half of the school takes place. Picture courtesy of the organisers.

15th to 27th July 2012
Rome, Italy

INSIS is a new international school aimed at providing a comprehensive training in the fundamental concepts of instrumentation for neutron scattering experiments.

The INSIS school is aimed at PhD students, postdoctoral researchers and early career scientists who wish to be involved in the design and development of novel instrumentation and components at present and future neutron sources. The course will provide basic and advanced lectures, simulation activities and tutorials about neutron scattering instrumentation.

The school is intended as a 'pipeline' initiative, to promote increased activities in instrument design and develop the next generation of instrument designers with a target audiaence of about 25 students. The school is composed of two distinct parts: the first week (15th– 20th July) will introduce basic instrumentation and components used in neutron scattering research; and the second week (22nd– 27th July) will provide a more in depth insight into neutron detector technologies. Students may participate in the first week, the second week, or both.

The deadline for applications will be the 31st of May 2012.

For more information please visit the website: http://neutrons.ornl.gov/conf/insis2012/

Laboratori Nazionali di Frascati, near Rome, where the school takes place. Picture courtesy of Laboratori Nazionali di Frascati

Next dates: 11th to 20th August 2012
Institut Montana Zugerberg, Zug & Paul Scherrer Institut, Villigen, Switzerland

Frequency: annual
The 'PSI Summer School on Condensed Matter Research' first took place in 2002, as a continuation to our traditional summer schools on neutron scattering, which first took place in 1993 in Zuoz/ Switzerland. The scope of the annual school is the training of Ph.D. and postdoctoral students in the experimental methods and probing tools provided by PSI to the scientific community: that is neutrons, muons and synchrotron light. Every year the school focuses on a specific topic, such as 'Functional Materials' (2009), 'Magnetic Phenomena' (2010) or 'Phase Transitions' (2011).

The programme of the schools includes lectures introducing basic theories and experimental techniques, mainly given by PSI staff, and comprehensive overviews of particular research fields mainly presented by international expert speakers. The lecturers are recruited internationally and are prominent researchers in their field, in Europe and worldwide. A poster session gives the participants the opportunity to present their scientific work.

In between lectures, participants have the chance to deepen their understanding of the methods and topics addressed through discussions with lecturers and PSI staff scientists.

Since 2009, the school is complemented by hands-on practical sessions carried out on instruments provided by SLS, SINQ and SmuS.
Twenty to thirty students are selected, divided in small groups, and introduced the experimental techniques with photons, neutrons and muons.

The PSI summer school is open to the Swiss and international scientific community and the language of the school is English.

The deadline for applications will be the 30th June 2012.

More information can be obtained on the website: www.psi.ch/summerschool

Students at the 2011 PSI summer school.
Picture courtesy of the organisers.

Jülich Centre for Neutron Science: Laboratory Course Neutron Scattering

Next dates: 3rd to 14th September 2012
Jülich and Garching, Germany

Frequency: annual
The JCNS Laboratory Course Neutron Scattering is organised annually since 1997 by Forschungszentrum Jülich .

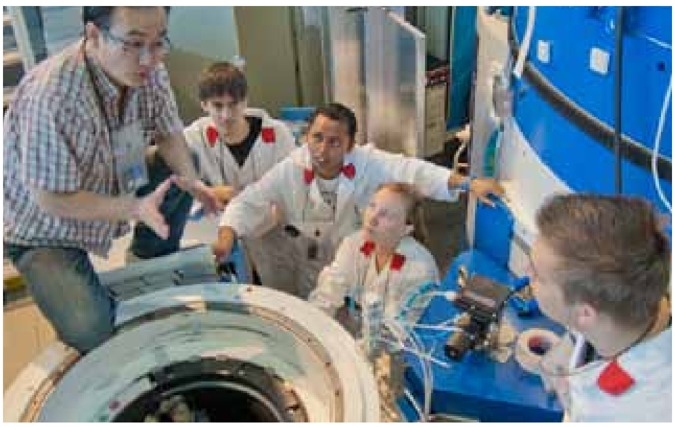
The aim of the course is to give students an insight into neutron scattering, the experimental technique and its scientific power. The laboratory course consists of one week of lectures held at Forschungszentrum Jülich followed by one week of neutron scattering experiments at the research reactor FRM II in Garching, Germany. The lectures include an overview of neutron sources as well as an introduction to scattering theory and instrumentation. In addition, selected topics of condensed matter science are presented. Students get to practice the topics addressed in the lectures through two hours of exercises per day. In the second week, 10–12 neutron scattering instruments are made available free of cost for five days. The participating students work in groups of five. Each group performs one neutron scattering experiment per day, so that each student experiences working on five different instruments.

In summary, the scientific programme of the laboratory course comprises 20 hours of lectures, 20 hours of exercises (half of the time in tutored groups), and 40 hours of experiments (including preparation and reporting). The number of students admitted is typically 50 to 60. The feedback collected from the students is consistently positive and the fact that many of the former participants are now scientists in neutron scattering-related workgroups shows that the course has a lasting success in education.

The whole course including local accommodation and meals is made available for free to the selected students. For non-German EU students, travel expenses are also reimbursed.

Deadline: 25th May 2012

For more information please see the website: www.neutronlab.de



Students at the 2011 Oxford school.
Picture courtesy of the organisers.

FAN du LLB

Next dates: 26th to 29th November 2012
Saclay, Gif sur Yvette, France

Frequency : annual
Fan du LLB is an annual school delivered in French and offering young French-speaking researchers a first contact with real experimental neutron scattering. The school is aimed at students and post-docs working in all scientific areas where neutrons can provide valuable insights, although priority is given to those having never had any contact with neutron scattering.

After an introduction to neutron sources and neutron scattering, ten different thematic subjects based on different scientific problems that can be addressed by neutron scattering, are proposed to the students. In groups of four to five, the students are then introduced to two different neutrons scattering techniques, during three days devoted to experiments and data analysis. One of the distinguishing features of the school is that the students often come with their own samples, which are tested during the training together with our demonstration samples. This ensures a good and efficient participation of the students. The course lasts for three and a half days.

The usual number of participants is 35.

For more information please visit the website : www-llb.cea.fr/fan

Students at the 2010 Fan du LLB school. Picture courtesy of the organisers.

Special Feature

Neutron imaging – past, present and future

Neutron imaging was first used successfully in 1935, shortly after the discovery of the neutron by James Chadwick. Since then it has developed and diversified in such a way that it is now used in research for a wide range of applications. Today, neutron imaging is at a pivotal point in its history. With the emergence of digital cameras and the rapid development of digital detectors with better spatial and temporal resolution, neutron imaging has developed into a valuable, reliable technique whose importance for materials research is finally being recognised. Many facilities around the world are dedicating beam lines to neutron imaging or building imaging instruments. NMI3 recently launch a Joint Research Activity in Neutron Imaging. In this article, Juliette Savin reviews the different neutron imaging methods available today, presents the new NMI3 Imaging Joint Research Activity and exposes some of the challenges still facing the technique.

What is neutron imaging?

Neutron imaging refers to a collection of non-destructive testing methods, which exploit the penetration of neutrons to investigate the internal structure of objects. According to the universal law of attenuation of radiation passing through matter, different materials attenuate neutrons differently. In a typical neutron radiography experiment, the object being investigated is placed in a well defined neutron beam. A 2D position-sensitive detector, often consisting of scintillator optically coupled to a CCD or EMCCD Camera, is placed on one side of the object and records the radiation transmitted through the object. The shadow image thus produced gives information about the internal structure of the object studied.

Whereas radiography results in a two-dimensional image of the sample studied, tomography allows to visualise samples in three dimensions. For neutron tomography, the sample rotates in the beam and multiple 2D radiography images are recorded. A 3D representation of the volume of the object can be reconstructed using a mathematical algorithm.

Few facilities in Europe offer access to instruments for radiography and tomography. At PSI´s NEUTRA, imaging is carried out thanks to SINQ´s beam of thermal neutrons while BEnSC´s CONRAD, FRM II´s ANTARES and PSI´s ICON offer cold neutron radiography and tomography. FRM II´s NECTAR makes use of the FRM II´s fission neutron spectrum. NRAD in Budapest uses BNC´s thermal neutrons.

Neutron imaging is complementary to other non-destructive imaging methods, in particular X-ray imaging. Whereas X-rays are scattered and absorbed by the electron cloud of an atom, neutrons interact with the atomic nuclei. Therefore neutrons are more sensitive than X-rays to light elements such as hydrogen, lithium, boron, carbon, and nitrogen. The degree of attenuation depends on the neutron energy. For some polycrystalline materials such as metals,

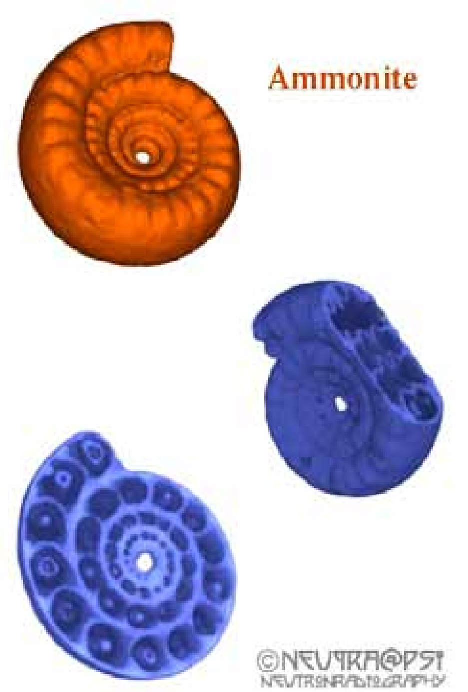


Figure 1: Neutron imaging can also be used in paleontology: neutron tomography of an ammonite. Picture PSI/NEUTRA.

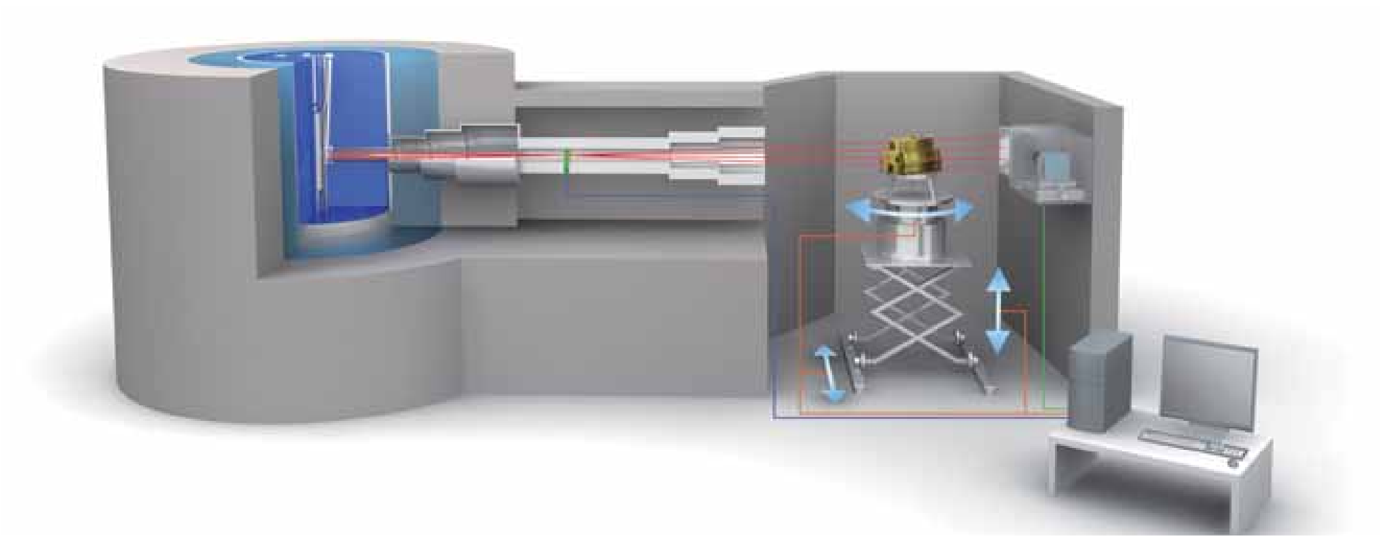


Figure 2: ANTARES, the cold neutron radiography and tomography station at FRM II. Picture courtesy of FRM II.

a strong energy dependency of the attenuation is observed in the cold neutron range, due to Bragg scattering from the crystal lattice.

Although neutron imaging cannot compete with X ray imaging in terms of resolution, the high penetration depth of neutrons allows to study large samples of some materials, with volumes of up to 100 cm^3 at medium resolutions, which X rays cannot do. This can be extremely useful in fields such as engineering.

“When you need imaging, you will usually start with X rays, because they are much easier to get” explains Thomas Bücherl from TUM, Germany. “But it is a good idea to have both, because you get information with X-rays that you do not get with neutrons, and vice versa.”

In some neutron imaging instruments, such as ANTARES at FRM II in Germany, there is the possibility to switch between neutron and X-ray beams to study the same samples.

Film versus digital

In the early days of neutron imaging, the technique was done with film, using gadolinium as a converter. This allowed only for a certain type of science to be carried out with neutron imaging and many criticised the fact that the method wasn´t a quantitative method.

“Film is not a digital system, it is not linear, and it is very difficult to get quantitative information out of it. You need to scan the film point by point” explains Burkhard Schillinger responsible for ANTARES at FRM II in Germany. According to him, this is why neutron imaging didn´t really make it in the neutron world before 1990.

Since the early 1990´s however, neutron imaging has been transformed by the digital revolution and the use of CCD cameras instead of film. Suddenly, pixels could be counted, grey values could be quantified and imaging became a quantitative method.

Researchers then realised that they could get much better imaging, with higher resolution, by sacrificing intensity for higher collimation of their neutron beams, since the new electronic detectors were much more sensitive than film. And this has now become the key factor in neutron imaging instruments - collimators define the Collimation ratio, the ratio of the distance between the sample and the collimator aperture over the diameter of the aperture. The intensity of the neutron beam inevitably drops with collimation but this can be compensated by using very sensitive detectors.

What applications for neutron imaging?

The unique ability of neutrons to easily penetrate metals and still be sensitive to light elements such as hydrogen, carbon, and nitrogen makes neutron imaging particularly useful for applications in fundamental research and industry.

For instance, neutron imaging is used routinely to highlight light materials such as hydrogenous substances with high contrast in engine parts or in hydrogen storing tanks and fuel cells.

Neutron imaging also allows to visualise the movement of fluids, such as oil or water, in large metal objects.

Neutron imaging has attracted the attention of companies in the car and aeronautical industries, which regularly visit neutron imaging facilities to carry out quality control tests or studies on engines, gear boxes or other metal parts.

Neutron imaging is non-destructive in nature, which also makes it an attractive method to analyse archaeological artefacts or pieces of Art.

Another application of imaging is the study of wooden objects, both for engineering or archaeological purposes.

Despite the wealth of applications of neutron imaging, neutron facilities have a hard time attracting users from the industry or archaeology worlds.

“Although they produce nice pictures, which can be attractive, our methods often seem too exotic to companies or museums” explains Eberhard Lehmann, Imaging group leader at PSI in Switzerland.

Users from outside who come to neutron facilities to do imaging often have problems interpreting the results they get with neutron imaging. “Very often users just leave the interpretation down to us, because they really don´t know what to do with their results” explains Anders Kästner, expert in imaging and ICON instrument responsible at PSI.

Looking to the future?

Despite the broad range of applications of neutron imaging, neutron imaging instruments have historically been given little space or importance within neutron facilities. “In a typical neutron facility, you´ll often find only one neutron imaging instrument” explains Dr Lehmann. And neutron imaging instruments need space, which is not always available at existing facilities to build new imaging instruments.

“For a long time neutron imaging wasn´t considered science. People thought: You´re just producing coloured images, that´s not science!” explains Burkhard Schillinger. Facilities didn´t want to dedicate beam lines to neutron imaging. But the digital era has dramatically transformed imaging and neutron imaging is experiencing a sort of new beginning, with facilities around the world suddenly investing in neutron imaging instruments.

According to Eberhard Lehmann, “there are indeed some good indications for an increase of the neutron imaging activities world-wide”.

Indeed, a neutron imaging and diffraction instrument for materials science, materials processing and engineering, IMAT, is currently in design phase at ISIS in the UK. In Norway, Kjeller is considering dedicating a beam line to neutron imaging, and one is under construction at IBR-2 in Dubna, Russia. In the Czech Republic, a test beam line is available at the Rez reactor. There are of course plans to build a neutron imaging facility at the ESS and the ILL in Grenoble is considering building a new neutron imaging facility.

Further afield, the reactor CARR in China already possesses two dedicated beam lines and ANSTO in Australia, J-PARC in JAPAN and SNS in the USA are planning their first neutron imaging facilities.

“These are really positive approaches and activities into the right direction” confirms Dr Lehmann, who is also president of the international Neutron Radiography society. All this suggests that the international neutron imaging community, which at the moment comprises around 200 people, excluding one-off users, is bound to grow. The community meets every two years alternately at the International Topical meeting on Neutron Radiography, which this year is taking place in Canada, and the World Conference on Neutron Radiography, which will be held in Switzerland in 2014.

New techniques

New, more sophisticated neutron imaging techniques have been developed recently, which also explains the new interest in neutron imaging. Techniques such as real-time imaging, stroboscopic imaging, energy selective imaging or neutron grating interferometry are now carried out routinely at facilities across Europe. They extend the scope of neutron imaging beyond conventional attenuation imaging and provide information about properties and phenomena until now difficult to study.

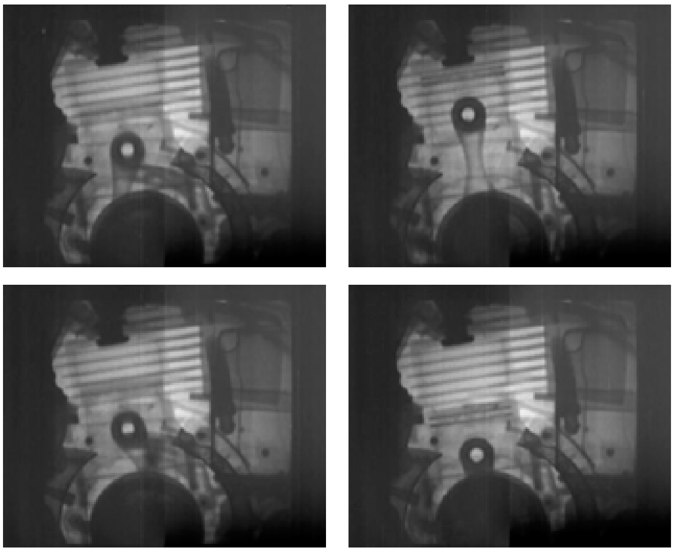


Figure 3: Stroboscopic imaging: frames from a two-stroke engine running at 3000 rpm. Pictures courtesy of Anders Kästner, ICON/PSI.

The development of highly-efficient detector systems allows for real-time imaging, which can be carried out at most neutron imaging facilities with an intensive beam. This technique allows to follow a dynamic process in real-time. The limited brilliance of today´s neutron sources only allows exposure times of a few milliseconds per image, but better time resolution can now be obtained with stroboscopic imaging.

The first stroboscopic imaging experiment was carried out in 2002 in collaboration between FRM II, the ILL, the University of Heidelberg and PSI. A four-piston BMW combustion engine was inspected by stroboscopic neutron radiography technique at the high flux test beam line H9 at ILL achieving exposure times of few hundred microseconds. Since then, stroboscopic imaging has proved to be ideal to visualize the dynamics of lubrification liquids and cooling agents in metal casing and has become the method of choice for the imaging of combustion engines in operation.

Neutron imaging can provide more accurate information on a sample if carried out with selected neutron energies. The proof of principle experiment for energy-selective imaging was carried out at ISIS. Use of this method requires a cold neutron spectrum. In this method, the transmission signal is recorded position sensitively depending on the neutron energy being varied. Due to Bragg scattering in the crystal lattice of the material in the sample, a diffraction contrast is observed in the transmission images. This contrast allows to distinguish between different crystallographic modifications of the material and hence to make phase separation in 3D, non-destructively. The diffraction contrast provides information about the presence of mechanical residual stresses and textures in the sample.

Neutron grating interferometry also requires a cold spectrum and it is not available everywhere. In Europe, two instruments provide this method as option to the users. ICON, the cold neutron imaging instrument at PSI, is one of the only instruments in Europe to offer energy selective imaging or neutron grating interferometry. A grating interferometric setup was recently was installed on the CONRAD beam line at HZB, in collaboration with PSI. Another grating interferometer will be installed at the upgraded ANTARES facility at FRM II soon. Grating interferomery can be used to investigate micro- and nano-heterogeneity of structures with sizes ranging from 0.1 to 10 µm.

Imaging of nano and magnetic structures

Better neutron imaging technology is still needed for imaging of very small structures and to improve the scope of magnetic imaging. Good, high-resolution magnetic imaging is crucial in particular for the study of superconducting materials.

NMI3 has decided to act on this by putting together a Joint Research Activity (JRA) focusing on the development of better components for neutron imaging and of new techniques using different interaction mechanisms of neutrons with matter to produce contrast, than conventional absorption imaging methods.

The JRA, funded by NMI3 and involving teams from FRMII, PSI, HZB, ISIS, LLB, JCNS and NPI, will focus in part on combining neutron imaging and scattering techniques, such as grating interferometry, which can be used to investigate structures at the micro and nano level together with innovative imaging with polarised neutrons, which can visualise magnetic fields in the direct space. The goal of this project will be the visualisation of microstructural changes in metallurgical samples under tensile or torsion stresses.

Another task within this JRA will be to develop techniques for vectorial magnetic imaging in the volume of nano and micro-structures. Existing magnetic imaging techniques (X-ray PEEM, AFM, electron holography) are mostly surface techniques and do not provide direct information about the volume magnetisation of nano-objects.

One of the main limitations in neutron imaging right now is the spatial resolution of the detectors available today. As part of the JRA, a team at FRM II will also work on the development of better detector components, for better high-resolution imaging.
The other limitation is the neutron flux available for experiments. "We should optimise our detectors and also our beams for neutron imaging experiments. At the moment we cannot compete with synchrotron radiation facilities, which have better flux and better intensities" explains Nikolay Kardjilov, coordinator of the Imaging JRA.
One way to optimise neutron beams is to improve optical components used to focus the beams, the aim of another Joint Research Activity supported by NMI3, the Neutron Optics JRA.

The future is looking bright for neutron imaging. If it wants to compete with other imaging techniques, more available, easier to carry out and to interpret, such as X rays, effort and funding for technical development will have to continue to flow.

For more information:
International Society for Neutron Radiology http://www.isnr.de/
7th International Topical Meeting on Neutron Radiography http://itmnr-7.com/
NMI3 Neutron Imaging pages: http://nmi3.eu/neutron-research/techniques-for-/imaging.html
PSI Neutron Imaging group : http://neutra.web.psi.ch/

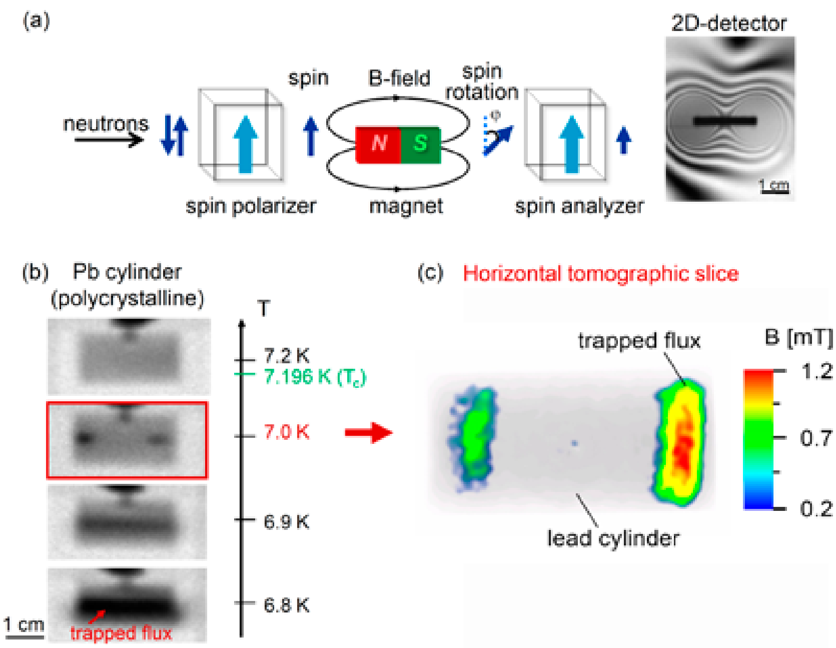


Figure 4. Imaging of magnetic materials and visualisation of magnetic fields (a) Incoming neutrons are spin-polarised, pass the magnetic field of a sample and go through a polarization analyser. A grey-scale image is generated after measurement by the 2D detector. (b) Temperature dependence of the residual magnetic field trapped in a piece of superconducting lead. (c) For a weak trapped residual field at T = 7.0 K, a tomographic investigation was done by rotating the sample around the vertical axis. A quantitative reconstruction of the magnitude of the magnetic field can be obtained. Images first published in Kardjilov, N., *et al.*, Nature Phys (2008) 4(5), 399. Reproduced with permission of the authors.

News from around the World

Welcome to the New ENSA Website!

By Emma Lythgoe and Javier Campo

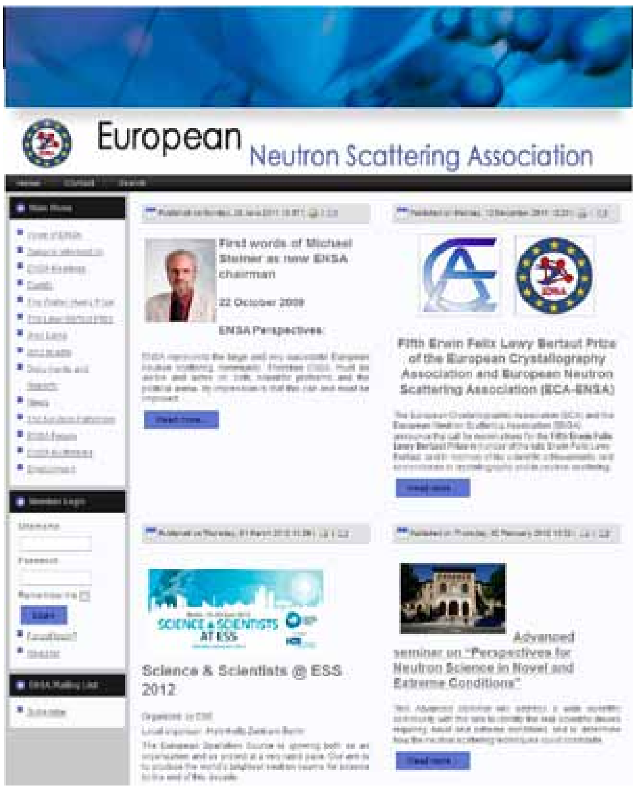
The European Neutron Scattering Association has launched a new web site devoted to the activities of the European Neutron Community.

Michael Steiner first stated in October 2009 that "ENSA represents the large and very successful European neutron scattering community. Therefore ENSA must be visible and active on both, scientific platforms and the political arena. My impression is that this can and must be improved". The aim of the ENSA website is therefore to assist with this vision, to improve the visibility of the European neutron scattering community as a whole as well as provide a communication channel for its members.

The new website provides not only general information about ENSA but also details of up and coming events and conferences. ENSA reports and documents are also available along with the latest news regarding the Walter Haelg and Lewy Bertaut Prizes. We would like to take this opportunity to invite you to visit us at http://www.neutrons-ensa.eu/ and take a look around. Register as a member to gain access to the ENSA documentation and join our distribution list to keep up to date with the latest news and employment opportunities.

We would also be delighted to receive any content of interest to the European neutron community and look forward to hearing your feedback.

The contact email address is:
info@neutrons-ensa.eu



Scientists gathered in Berlin to define future ESS science

Around 340 scientists gathered on 19-20 April in Berlin for the Science & Scientists at ESS meeting to discuss the technical challenges neutron science faces and the scientific perspectives of the future European Spallation Source (ESS).

During two days, about 25 plenary talks, 25 oral contributions in parallel sessions and 100 posters were presented illustrating the science the European user community would like to do in the future and the instrumental possibilities needed to do so.
First on the agenda were presentations on the progress made in designing the ESS. When built in 2019, the European Spallation Source will be one of Europe's largest science facilities. Currently, 17 European countries are planning to build the ESS facility. Many partners within those countries participate in collaborations and in-kind design of the ESS. Their work was presented mainly in the poster and parallel sessions. This aspect of the program was completed with presentations on innovative neutron technology.

Second in the program were plenary scientific talks presenting science drivers in the fields of quantum matter, material science, soft matter, biology and fundamental physics. Those talks were complemented by shorter ones on the findings of the ESS Science Symposia organized in recent months all over Europe. The various topics included food science, engineering and thin films research. Through these reports the opinions and ideas of a large user community involving several hundreds of scientists were channeled back into the project. "The conference has shown an impressive breadth in scientific and experimental ideas", says Dimitri Argyriou, ESS Science Director. "We have taken part of many, many scientific topics and valuable, innovative ideas that will be fed into the ESS science planning."
The third and most important aspect was discussing the instrumental requirements for ESS and how they can be realised. The ideas were first presented by ESS and its partners during the poster exhibition. Afterwards parallel sessions on the instrument classes were organised in which the scientists could express their specific needs to perform their future science program and the instrumental performance parameters required. "I am very happy to see that such a large number of scientists are keen to contribute to building experimental opportunities and scientific communities around the ESS, especially considering the very early stage in the ESS project. Scientists are not only waiting passively for ESS to open in 2019, but are engaging already now", says Dimitri Argyriou.

Another highlight of the meeting was the evening lecture by Gerry Lander on "Discoveries that Changed the World". Focusing on the life of Lise Meitner and other scientists during the early 20th century, he established a fabulous link between neutron science and the city of Berlin, already at the time a center for frontier science.

With one of the world's largest neutron user community, Germany has a particular interest in the ESS project. Several German research institutes, universities and laboratories are taking active part in the design and construction of ESS. In this, Berlin was the perfect venue for this ESS Science & Scientists meeting. The meeting was initiated and organised by ESS, and thanks to the local organisers at Helmholtz-Zentrum Berlin für Materialien und Energie it was perfectly hosted at a beautiful venue.
To find out more and download additional material, visit the website www.esss.se/s-and-s.

Picture: ESS AB

Coordination and Management

Kicking-off NMI3-II

NMI3 partners meet at ILL in Grenoble for the start of the new NMI3 project in FP7

ILL, the coordinating facility of NMI3, recently hosted the meeting that set the start of the new NMI3 project, NMI3-II, which will run from 2012 until 2016. The meeting brought together representatives of all partner institutions, as well as Access programme coordinators from all member facilities, the coordination team and members of the Joint Research Activities funded by NMI3.

During the General Assembly, Access programme coordinators from all facilities presented their facility and the instruments and services they offer. The new Joint Research Activities in Imaging, Detector Development, Muons, Tools for soft- and bio-materials and Advanced Methods and Techniques were also presented and more information about them can now be found on the Joint Research Activities pages on our website.

During a board meeting that took place before the kick-off meeting, Mark Johnson from ILL was appointed coordinator of NMI3-II. Helmut Schober will coordinate NMI3-I until it ends in January 2013.

Participants of the NMI3 Kick-off meeting in 2012. Picture: Serge Claisse, ILL

Calendar - upcoming meetings

General assembly	5th and 6th December 2012	FRM II, Garching, Germany

Please note: JRAs have the possibility to organise a meeting at FRM II.

Picture: NMI3

Winners of the second Illustrating NMI3 picture competition

We are pleased to announce the winners of the 2nd ´Illustrating NMI3´ picture competition.

The first prize goes to Elisabeth Blackburn from the University of Birmingham in the UK, for her picture of an external vacuum chamber attached for rapid in situ sample change on the Birmingham 17 T cryomagnet.

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.

The second prize goes to Holger Kohlmann from Universität des Saarlandes in Germany for his picture of a Sapphire gas pressure cell, on the high-intensity diffractometer D20 at the Institut Laue Langevin (ILL).

Sapphire gas pressure cell for real time in situ neutron powder diffraction of solid-gas reactions with hydrogen under high pressure on the high-intensity diffractometer D20 (ILL). The sample inside the sapphire single crystal is heated by two laser beams guided through the optics seen on the left and righthand side.

The third prize goes to Peter Linder for his picture of the new 40m long detector tank of the small angle neutron scattering (SANS) instrument D11, at ILL. The picture celebrates the 40 years of D11.

40 years D11, the unique small angle neutron scattering (SANS) instrument: view of the new 40m long detector tank.

Three more pictures were selected as fourth-prize winners, to be displayed on the NMI3 website:

Alexis Chezière for his picture of a colleague on the three-axis spectrometer IN1 at ILL.

Nico Grimm from Helmholtz-Zentrum Berlin (HZB) beautifully photographed a high precision temperature controlled sample cell.

Andrew Sanozov´s photograph of the new very intense polarized neutron diffractometer VIP on the hot source of the Orphée reactor, Laboratoire Léon Brillouin (LLB) also wins a fourth prize.

Please visit www.nmi3.eu to view these pictures.

Editors

Juliette Savin	NMI3 Information Manager
Jürgen Neuhaus	NMI3 Networking Manager
Mark Johnson	NMI3 Coordinator
Miriam Förster	NMI3 Project Manager

Design and Layout

Ramona Bucher	FRM II

Contact

Technische Universität München
Forschungs-Neutronenquelle
Heinz Maier-Leibnitz (FRMII)
Lichtenbergstraße 1
85748 Garching
Germany

Phone	+49(0)89 289 14615
E-Mail	info@nmi3.eu
Web	www.nmi3.eu