Jülich Centre for Neutron Science
A leading role in neutron science

Research with neutrons, with its interdisciplinary approach, is indispensable for modern science. It makes a major contribution to solving the grand challenges facing mankind in areas such as the environment, energy, key technologies and the life sciences.

Neutron research requires large infrastructures to produce and use neutrons – research reactors or spallation sources – administered and supported by a distinct organisation to make the most efficient use of such facilities. The Jülich Centre for Neutron Science (JCNS) is a major user-access platform. It provides access to unique experimental facilities for the vibrant German and international user communities, and promotes the development of neutron techniques at the highest level.

In order to enhance the scientific quality of the user experiments, JCNS backs the offered instruments with large in-house scientific research activities covering the fields of highly correlated electron systems and magnetism as well as soft matter and biophysics. This important in-house expertise is available for users who want to join forces and cooperate. In this context also complementary techniques such as cryo electron microscopy, dynamic and static light scattering, MBE-facilities for in-situ thin layer preparation to name a few offered.

JCNS is also exporting the fruits of its scientific experience and knowledge beyond European borders. It supports the development of neutron scattering methods in China through the recent transfer of instruments from the former DIDO reactor of Forschungszentrum Jülich to the China Advanced Research Reactor CARR. Such initiatives will help strengthen scientific links between the two countries.

A future in Europe

Finally, a major goal at JCNS is preparing the future of neutron research in Europe by leading the German contribution to the Design Update study of the European Spallation Source ESS, to be built in Lund, Sweden. This facility will lead the world in providing neutron beams with unprecedented intensities that will revolutionise many areas of research, particularly in the biological area. JCNS is ready to contribute its expertise in instrumentation and user-operation – gained from its activities at the SNS spallation source in the US – to this future, next-generation neutron source.

Professor Dr. Dieter Richter
Scientific JCNS Director 2012

Professor Dr. Thomas Brückel
Managing JCNS Director 2012
The Jülich Centre for Neutron Science (JCNS) at the Forschungszentrum Jülich provides access to state-of-the-art experimental facilities in neutron science for both German and international user communities. It operates instruments at leading national and international sources.
The main national centre is the continuous neutron source in Garching Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), operated through a cooperative agreement between the Technical University Munich and the Helmholtz centres led by Jülich. Outside Germany, at the high-flux reactor of the Institut Laue-Langevin (ILL) in Grenoble, JCNS operates instruments jointly with the CEA in Grenoble through the ILL’s Collaborative Research Group scheme. In the US, JCNS is responsible for instruments at the world’s first megawatt-class (accelerator-based) spallation source, SNS, in Oak Ridge, Tennessee. These instruments are made available to users through an independent peer-review process.

JCNS also carries out a globally-recognised development programme for neutron methods and instruments. These include the development of:

- cutting-edge neutron instrumentation;
- specialised sample environments;
- data reduction and modelling software.

Much of this development is driven by the research that JCNS carries out in two areas of major scientific and technological importance that depend heavily on neutron-scattering methods: research into complex soft matter, including biological systems; and advanced magnetic and electronic materials. In addition, JCNS provides the infrastructure for users of neutrons: dedicated laboratory facilities for preparing and characterising samples, and a team of expert neutron specialists.

JCNS also leads the way in Germany in exploiting new opportunities for neutron scattering science being developed at the next generation of neutron facilities.
Snapshots from 2009–2011

€ 1.1 Million in EU funding for Jülich neutron research

April 2009:
Neutron researchers at Forschungszentrum Jülich have secured funding worth around € 1.1 million as part of the 7th EU Framework Programme for the promotion and advancement of neutron scattering in European science. With this funding, JCNS is able to offer European neutron researchers in science and industry another 102 measuring days with Jülich research instruments at the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II). A total of € 450,000 will be spent on the development of new neutron scattering methods. To this end, JCNS plans to coordinate a development network in which seven European research groups will work to further develop possible applications of polarised neutrons for the investigation of magnetic properties in solid state physics or the dynamics of individual molecules in polymer physics and biology. The funding was backdated to 1 February 2009 and covers a period of four years.

Bavarian Premier visits neutron researchers

July 2010:
On his visit to the neutron source Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II) in Garching, Germany, the Bavarian Premier, Horst Seehofer (second from left) made a tour of the neutron guide hall together with Prof. Sebastian M. Schmidt (second from right), member of the Jülich Board of Directors. The guide hall housed eight instruments belonging to the Forschungszentrum at the time of the visit. (From left: Dr. Wolfgang Heubisch, Science Minister of Bavaria, Bavarian Premier Horst Seehofer, Prof. Dr. Wolfgang Herrmann, President of TUM (Technische Universität München), Prof. Dr. Winfried Petry, Scientific Director of the FRM II, Prof. Dr. Sebastian M. Schmidt, Member of the Board of Directors of Forschungszentrum Jülich and Dr. Klaus Seebach Administrative Director of FRM II.)
BMBF supports Jülich’s contribution to the European Spallation Source ESS

November 2010:
Forschungszentrum Jülich has secured approximately € 6.4 million from the German Federal Ministry of Education and Research (BMBF) in support of its contribution to the planning phase of the European Spallation Source ESS in Lund, Sweden. Thomas Rachel (left), MdB, Parliamentary State Secretary at the German Federal Ministry of Education and Research, presented the certificate of funding approval to Prof. Sebastian M. Schmidt, member of the Board of Directors at Forschungszentrum Jülich and coordinator of the German contribution to the ESS. The BMBF will support the German research facilities and universities taking part in the planning phase with an initial sum of € 15 million.

JCNS intensifies collaboration with neutron source FRM II

December 2010:
Neutron research in Germany receives significant support: Forschungszentrum Jülich, the Technische Universität München (TUM), the Bavarian Ministry of Science and the Federal Ministry of Education and Research (BMBF) have been successful in regulating, in a single, encompassing cooperation agreement, how Germany’s national research centres, with a budget of around € 300 million, plan to improve the way in which the most powerful German neutron source, the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), will be used in scientific research from 2011 to 2020. The national research centres in the Helmholtz Association agreed to invest approximately € 30.3 million per year in neutron research. Forschungszentrum Jülich aims to expand its range of scientific instruments to eleven devices by 2013 to maintain its position at the cutting edge of research. A total of 30 large instruments will then be available at FRM II. The research centres themselves contribute around € 10.5 million per year from their budgets; the BMBF supports the centres along with the scientific infrastructure of the FRM II with a further € 19.8 million per year, of which 16.7 million derives from special funding and 3.1 million from research consortium sponsorship.

JCNS researchers benefit from powerful neutron source

February 2011:
During his trip to China, Prof. Sebastian M. Schmidt (left), member of the Board of Directors of Forschungszentrum Jülich, visited the CARR research reactor in Fang Shan near Beijing, where physicists and engineers from JCNS have installed two single-crystal diffractometers and one triple-axis-spectrometer. With its power output of 60 megawatts, CARR will rank as one of the most modern research neutron sources in the world. Scientists from JCNS will be able to use this powerful neutron source for their experiments; Jülich is entitled to 30 % of the beam time until 2017 and is a member of the committee to decide on how a further 30 % will be assigned.
Neutrons are subatomic particles that form part of the nucleus, and are produced in various nuclear reactions including fission. In the past half century or so, they have provided an increasingly important set of analytical methods that allow scientists to see deep into the heart of matter down to the molecular and atomic scale, and beyond.

How does neutron scattering work? Because neutrons are microscopic objects, they obey the laws of quantum mechanics and behave both as particles and waves. Like X-rays, their wavelength range (which is linked to their energy) matches typical distances between nearby atoms or molecules in a material. Thus, when neutrons are scattered, for example, by regular arrays of atoms in a crystal, they reveal their atomic arrangement. The reflected neutron waves interfere with each other – either enhancing their intensity or cancelling it out – to produce a characteristic diffraction pattern that contains information about the structure.

In addition, some of the neutron energy may be exchanged with the atoms or molecules as a result of their motions, which can also be measured. Inelastic neutron scattering is an invaluable probe of atomic and molecular dynamics.

Special properties of neutrons
Neutron and X-ray methods provide complementary information by the very nature of their interactions, so researchers often use both techniques in the same experimental project. While X-rays interact with the electron clouds in atoms, neutrons impinge directly on the atomic nuclei, and can readily detect light atoms such as hydrogen, which are not easily seen by X-rays.

Isotopes of the same element may scatter quite differently for example, hydrogen and deuterium have different scattering strengths. Substituting deuterium for hydrogen – deuteration – in a selected part of a molecule or molecular assembly provides contrast and allows that component to be highlighted. Furthermore, solvents can be partially deuterated such that they have the same scattering strength as one part of a dissolved molecule, rendering it invisible, so that only another part of the molecule is seen.

This technique of contrast matching can offer advantages when applied in other contexts. Neutrons penetrate matter easily but do not damage delicate structures such as those found in biological materials. They are able to probe complex macromolecular and biomolecular structures, and their realistic behaviour in a watery environment is similar to that found in living cells.

Neutrons also have another property that makes them a very powerful probe, that of a magnetic moment or spin. This means that they can interact with single, unpaired electron spins in materials, which are associated with magnetism.

Neutron methods
Today, neutron techniques have expanded, and can explore the structure and behaviour of a wide range of materials. Neutron diffraction was first applied to samples that were large, single crystals. Today, with the aid of computer methods, neutron scattering can be used to study powders, solutions and thin layers, and even large engineering components. Specialised instruments are continually being developed to extract information at a particular length-scale or energy-range, from a wide variety of sample types over a range of physical conditions such as temperature and pressure.
The benefits of neutron

These include:

- **Small angle neutron scattering (SANS)** in which a very long spectrometer detects scattering at small angles that are indicative of large interatomic distances of hundreds of nanometres. SANS is crucially used to study large molecules and polymers, and self-organised molecular assemblies found in biological systems.

- **Powder diffraction** is useful for analysing materials that are not available as a single crystal. It is often difficult to grow large crystals of some metal compounds; ceramics and geological materials often have a multicrystalline structure.

- **Reflectometry** is an approach in which neutrons are bounced off thin layers or interfaces usually on supported surfaces. It is successfully used to look at molecular assemblies that model biological cell membranes.

- **Polarised neutrons** – neutrons with a defined spin direction – can detect the orientations of magnetic spins in materials, and are often applied to studies of exotic types of magnetism.

- **Inelastic scattering** spectrometers are designed to cover dynamics over a range of time-scales that also correspond to particular length-scales. Time-of-flight (TOF) spectrometers determine the change in energy by measuring the time taken for the scattered neutrons to reach the detector; they detect fast dynamics of less than one-tenth of a nanosecond. Neutron backscattering spectrometers measure neutrons reflected virtually backwards, which relay information on the energy changes associated with nanosecond motions. Another ingenious technique, neutron spin echo (NSE), detects very low-energy processes happening on the microsecond scale. It is similar to TOF, but employs a combination of polarised neutrons and magnetic fields to detect small energy changes in the sample, which result in subtle alterations to the polarisation states of the scattered neutrons.

**Neutron production**

All these techniques require intense, carefully tailored beams of neutrons, which are produced either in a nuclear reactor, or by the process of spallation whereby neutrons are released from a target when an accelerated beam of protons strikes it. This requires, therefore, a central facility dedicated to the production and use of neutrons.

Applications

Neutron scattering experiments now underpin research in many areas of technological and societal importance:

- **Soft materials** – design of plastics, reinforced composites, cleaning products, cosmetics and food formulation

- **Biomedicine** – biomolecular processes involving cell membranes, proteins and other macromolecules, drug delivery and gene therapy

- **Electronics** – new physics leading to advanced electronic and magnetic materials and devices for computer processing, data storage and sensors

- **Green energy** – development of efficient catalysts, batteries, fuel cells, hydrogen-storage materials, and superconducting power transmission

- **Nanotechnology** – design of electronic and medical devices working at the molecular level

- **Engineering** – studies of advanced ceramics and alloys, and wear and tear detection

- **Geology and environment** – processes involving oil prospecting, waste management and soil remediation

- **Art, archaeology and palaeontology** – non-destructive analysis of artefacts
Scientific highlights

Magnetism and superconductivity

Neutrons are ideal probes for the investigation of magnetic properties. JCNS instruments help to reveal the secrets of high temperature superconductivity or to find innovative materials for information storage.
Neutron studies on a cerium-based metal alloy reveal the quantum critical role of magnetism in turning the material into a superconductor at ultra-low temperatures.

Oliver Stockert

Unconventional superconductivity

Just over a century ago, in 1911, Heike Kamerlingh Onnes showed that mercury conducted electricity without resistance when cooled with liquid helium to 4.2 K. Further low-temperature superconducting materials – metal alloys – were subsequently discovered. The phenomenon was eventually explained satisfactorily in quantum terms as a current of electrons coupled together in so-called Cooper pairs. An ensemble of Cooper pairs could surf through the compound’s crystal lattice unimpeded, by interacting through quantum vibrations (phonons) of the lattice.

Then in 1986, a new class of superconductors was discovered, based on mixed metal copper oxides, cuprates. They became superconducting at much higher transition temperatures (Tcs), going up to 133 K. The cuprates have since been joined by another series of metal compounds, the iron-based pnictides, which also have higher Tcs than most conventional superconductors. Even early on after their discovery, it became obvious that the mechanism for superconductivity in these materials was different, and appeared not to involve phonons. Instead, evidence started to emerge that the Cooper pairs resulted from cooperative, magnetically-driven interactions in the form of ‘spin density wave’ fluctuations. In some metallic compounds, the electron spins of any magnetic atoms in the structure align antiferromagnetically (spins in opposite directions) setting up moving waves of spin density that are not tethered to the crystal lattice. The exact mechanisms involved are still not well understood, and are the subject of intense theoretical and experimental research.

Heavy fermion compounds

One group of exotic conducting materials called heavy fermion compounds is now providing a promising probe into the whole area of unconventional superconductivity. At low temperatures in certain metallic alloys, the charge carriers responsible for electrical conduction are not bare electrons but composite quasi-particles that move slowly, as though they have a mass of up to 1000 times greater than normal electrons. Heavy fermion compounds incorporate rare-earth or actinide elements characterised by complex electronic structures with outer ‘f’ electrons that appear to interact with the conduction electrons. They were discovered as early as 1975, and four years later – indeed, seven years before the discovery of the cuprates – the first superconducting version was found, cerium copper silicide (CeCu₂Si₂); the Tc was ultra-low, however, at 0.6 K.

It was clear, even then, that the superconductivity was unusual. The cerium f electrons interact antiferromagnetically, and it seemed likely that this magnetic response mediated the formation of the Cooper pairs, just as in the cuprates. Furthermore, as with other unconventional superconductors, slightly changing the proportions of the constituent elements, introducing new elements into the structure, or increasing the pressure, could tip the compound from a normal into the superconducting state. For example,
increasing the ratio of copper to silicon in CeCu$_2$Si$_2$ turns it into a superconductor. It appears that it is the process of bringing the magnetic atoms closer together that triggers the superconductivity.

Another important concept that specifically comes into play in heavy fermion compounds is that of the quantum critical point (QCP), which marks a set of physical conditions in which subtle quantum energy fluctuations arising from Heisenberg’s Uncertainty Principle mediate a transition between two physical phases that are very close in energy. In the case of CeCu$_2$Si$_2$ they are the antiferromagnetically ordered state and the nonmagnetic (paramagnetic) ground state, with the appearance of superconductivity around the QCP. Quantum critical fluctuations manifest themselves at absolute zero temperature or close to it, when classical thermal effects have subsided. It was suggested that quantum fluctuations in magnetic energy – tiny antiferromagnetic excitations close to absolute zero – were responsible for bringing about the formation of Cooper pairs and so driving the superconducting transition in CeCu$_2$Si$_2$.

**Probing quantum fluctuations**
The technique of inelastic neutron scattering provides the ideal tool for investigating this idea because it can detect very
Magnetic clues to an unusual superconductor

Small changes in the magnetic energy states of a material. However, until a few years ago, detailed studies of CeCu$_2$Si$_2$ were hampered not only by the difficult challenge of growing crystals large enough for neutron scattering experiments, but also by the lack of technologies needed to detect very weak magnetic effects at the required millikelvin temperatures, i.e. close to absolute zero. Today, better instrumentation has allowed us to re-visit this well-studied material, and use neutron methods to probe in detail how the antiferromagnetic state evolves into the superconducting state via quantum critical magnetic fluctuations.

Using the cold-neutron triple-axis spectrometer, IN12, at the ILL, in combination with a dilution fridge, we first carried out inelastic measurements on CeCu$_2$Si$_2$ over a range of temperatures above and below the $T_c$. We were able to obtain energy spectra of the magnetic excitations associated with the spin fluctuations. We noted that they slowed down considerably on approaching the $T_c$, when a gap in the spin excitation spectrum opened up, signifying that a threshold energy is needed to break up the Cooper pairs. Other types of experiments had measured the superconducting condensation energy, which we were able to compare with the magnetic energy changes. We found that the magnetic energy reduction was more than 10 times that of the condensation energy, as is also found in the cuprates, and concluded that the antiferromagnetic fluctuations associated with a QCP are strong enough to drive the superconducting transition.

We then carried out further inelastic neutron scattering experiments on CeCu$_2$Si$_2$ below the $T_c$ to look at the magnetic behaviour as close to the QCP as possible, and so further scrutinise the role of quantum critical fluctuations. This involved suppressing the superconductivity by applying a magnetic field, and scanning the magnetic excitation spectrum down to 0.05 K. We found that the lifetime of the fluctuations got longer and longer such that a state of permanent long-range antiferromagnetism is almost reached, showing the closeness in energy between this state and the superconducting state. The scaling of these spin fluctuations conformed to theoretical predictions that suggested the presence of a spin density wave instability. While in the cuprates and pnictides, the spin fluctuations are two-dimensional as expected with their layered structures, in the heavy fermion compound CeCu$_2$Si$_2$ they are truly three-dimensional.

This is the first time that quantum critical magnetic fluctuations associated with a QCP have been traced across the $T_c$ in this way. The results help to provide a better understanding of all materials that exhibit unconventional superconductivity, and thus aid the search for the Holy Grail of a material that is superconducting at room temperature.

References


Oliver Stockert

is a senior scientist at the Max Planck Institute for Chemical Physics of Solids in Dresden, Germany.

Inelastic neutron scattering spectra of CeCu$_2$Si$_2$ taken in the normal state (black) and the superconducting state (red) indicating the opening of a magnetic excitation gap in the superconducting state at the antiferromagnetic wave vector Q$_{AF}$. Dashed lines show the magnetic signal while the solid lines are the sum of the magnetic signal and the background contribution.

Inverse lifetime of the spin fluctuations versus temperature in CeCu$_2$Si$_2$ (data taken in the normal state at Q$_{AF}$ and in magnetic field of B = 1.7 T to fully suppress superconductivity). The solid line denotes a fit to the data as expected for a spin density wave instability. Upon lowering the temperature the lifetime gets longer and longer.
During the past quarter of a century, one of the most exciting scientific advances has been the discovery and study of compounds that conduct electricity without resistance at the relatively high temperatures of liquid nitrogen and above. These superconductors not only offer a practical route to providing power without energy loss, as well as novel technologies based on their unusual properties, they have also opened up a new frontier in our theoretical understanding of complex electronic behaviour in the solid state.

The first high-temperature superconductors discovered were mixed-metal copper-oxide compounds – cuprates – which have layered structures. The underlying mechanism for the phenomenon has proved to be different from that in conventional, low-temperature superconductors.

Unconventional superconductivity

Magnetism in the family of pnictide superconductors

Yinguo Xiao

Neutron studies of a europium iron arsenide compound reveal subtle relationships between its crystal structure and magnetic behaviour that are helping researchers understand high-temperature superconductivity.
Magnetism in the family of pnictide superconductors

It appears to be linked to magnetism, in particular, antiferromagnetism in the copper-oxide layers, but exactly how this occurs is still not well understood. From the outset, neutron-scattering techniques have played a crucial role in trying to elucidate the mechanism underlying high-temperature superconductivity.

Iron-based superconductors
Recently, a new family of high-temperature superconductors was discovered, based on iron arsenide compounds – pnictides – that share some similarities with cuprates. Most of the research on pnictide superconductors has focused on two structure types, RFeAsO1−xFx – where R is a rare-earth element, and AFe2As2 – with A being an alkaline-earth element such as barium (Ba⁺⁺), calcium (Ca⁺⁺) or strontium (Sr⁺⁺). They are known as the 1111 and the 122 families respectively. They, too, have layered structures, with one iron-arsenide layer per unit crystal cell in the 1111 compounds, and two such layers in the 122 versions. It is these layers that are responsible for the superconductivity.

The pnictide compounds demonstrate an even closer interaction between superconducting and magnetic properties, with the iron atoms ordering antiferromagnetically when cooled below a certain temperature. The onset of this order is accompanied by a change in crystal structure from tetragonal to orthorhombic (T–O phase transition) in the 122 family, but is preceded by the T–O transition for the 1111 family. This magnetic order is then suppressed by introducing ions that can donate or accept electrons, i.e. can introduce electrons or ‘holes’ into the unit crystal structure (a process known as doping). This changes the electronic behaviour such that superconductivity starts to emerge instead, reaching a maximum value for a given level of doping. This is similar behaviour to that of cuprates – although in the case of the iron-based compounds, the transition to the superconducting state (Tc) is at somewhat lower temperatures - up to 56 K. However, in some pnictides, superconductivity and magnetism are not mutually exclusive; there is actual overlap between the superconducting and magnetic states.

In order to understand this unconventional superconductivity better, physicists have been probing this relationship by looking more closely at magnetic behaviour in the pnictides. We decided to look at the magnetism in an unusual 122 pnictide containing the rare earth, europium, as the ‘A’ ion, EuFe2As2. Europium ions have a large magnetic moment, and investigating the interplay between the europium and iron magnetism may provide a deeper understanding of the physical properties of iron pnictides.

Superconductivity can be introduced into the compound by gradually replacing the europium ions (Eu²⁺) with alkal metal ions such as sodium (Na⁺) or potassium (K⁺), or by replacing arsenic with phosphorus. The Tc is observed to be 31 K and 26 K for Eu₀.5K₀.5Fe₂As₂ and EuFe₂(As₀.8P₀.2)₂ respectively. In the latter, superconductivity and magnetism actually coexist, as the superconducting transition is followed by magnetic ordering of the europium ions. Superconductivity can also be introduced by applying pressure to the parent compound.
We first analysed the magnetic structure using single-crystal neutron diffraction on the hot-neutron four-circle diffractometer HEIDI at the neutron source Forschungs-Neutrenquelle Heinz Maier-Leibnitz (FRM II). The diffraction data were collected at various temperatures from 300 K down to 2.5 K, which is well below the temperatures at which both iron and europium ions reportedly order. We could also follow both the structural and magnetic changes with decreasing temperature, through the T–O transition, which happens at 190 K.

As with other 122 pnictides, the structural changes at the T–O transition are both complicated and significant, due to the fact that two of the crystal axes become interchangeable when the orthorhombic state is entered, allowing the crystal to reconfigure itself in two different directions simultaneously, resulting in a so-called twinned structure. This means that the diffraction pattern is split into multiple peaks.

The T–O transition and the onset of antiferromagnetism does indeed happen at the same temperature, indicating that the magnetic behaviour is affected by the crystal structure in these compounds. Below 19 K, the europium ions order antiferromagnetically, which is in good agreement with previous electronic and magnetic measurements. There was no detectable change in the magnetic ordering of the iron at this transition, which suggests that the magnetic interactions between the iron and europium ions are rather weak.

No magnetic gain, no strain

We then went on to use neutron diffraction to investigate the effect of a magnetic field on the same material. Using the thermal neutron two-axis diffractometer, D23, at the ILL, we followed the evolution of magnetic order under magnetic fields up to 3.5 tesla over a range of temperatures down to 2 K.

We found that above a certain magnetic field strength, which also depended on temperature, the europium ions change...
from an antiferromagnetic to a ferromagnetic alignment along the direction of the applied field. What is particularly interesting is that the magnetic field appears to introduce a considerable mechanical strain in the twinned crystal structure, and, when strong enough, causes the crystal axes to re-align to form a single de-twinned structure. This realignment reverses back to the twinned structure as the field is reduced, which suggests the existence of magnetic shape-memory effect.

We noted that the critical field at which the de-twinning occurs is closely correlated with the field that induces the ferromagnetic transition of the europium ions. The excess energy supplied by the applied magnetic field to the ferromagnetic europium ions is taken up by the crystal lattice, resulting in mechanical movement. Moreover, in-plane magnetotransport properties of EuFe$_2$As$_2$ were also investigated using angle-dependent magneto-resistance measurements. We observed a strong anisotropy in the magnetotransport properties, and there is an intimate correlation with the ordering states of both the europium and iron spins, which provides direct evidence of the coupling between the conduction electrons and the ordered spins.

There is clearly a strong coupling between spin, charge and lattice in superconductor parent compound EuFe$_2$As$_2$. We are carrying out further experiments to study the effects of doping and applied pressure, in the hope of disentangling further this fascinating and complex set of behaviours.

**References**


Unconventional superconductivity

What makes a superconductor?

Dirk Johrendt

A new group of iron-based compounds demonstrating superconductivity shows some puzzling magnetic behaviour. Their exact composition also plays a key role in influencing the subtle electronic properties, but is difficult to characterise. Neutron powder diffraction provides the best way to analyse their structures and magnetic behaviour, and determine how and when superconductivity arises.

The discovery of the pnictide superconductors, containing layers of iron bound to arsenic (or phosphorus), has opened a new chapter in understanding high-temperature superconductivity. It is widely accepted that it is the weak magnetism within the iron layers that plays the critical role in harnessing the phenomenon.

Several families of pnictides containing superconducting members have now been discovered. Their crystal structures include combinations of the ions of different metals, particularly the transition metals and rare earths whose complex electronic structures generate a rich variety of physical and structural phenomena. If the ions are chemically similar (i.e. have the same size and/or electronic charge, or valency) – especially if they can substitute for the iron atoms – they can modulate the electronic and magnetic behaviour in a significant way.

In most pnictides, superconductivity occurs when magnetism is suppressed by substituting ‘alien’ ions (doping), or by applying pressure, which changes the interatomic distances within the crystal structure. Modifying the composition slightly can alter the subsequent crystallographic and electronic configuration, and thus may have a profound effect on the magnetic or electronic correlations triggering superconductivity. It is extremely difficult to develop a rigorous theoretical picture of the underlying mechanisms, so it is not surprising that the experimental investigation of the electronic and magnetic behaviour in pnictide variants is an extremely active area of research.

A new family

While much research has been carried out on the pnictide 122 family, AFe$_2$As$_2$, containing two adjacent iron-arsenide layers separated by alkaline-earth atoms, pnictides with structures containing iron-arsenide layers separated by thicker layers of mixed metal oxides also show reasonably high superconducting transition temperatures ($T_c$s). They include the new family of ‘21311’ compounds, Sr$_2$MO$_3$FeAs – where M is, for example, vanadium ($V^{3+/4+}$), scandium ($Sc^{3+}$) or chromium ($Cr^{3+}$). The vanadium compound, Sr$_2$VO$_3$FeAs, has a $T_c$ of 37 K, demonstrating the potential of such compounds. The analogous chromium compound, Sr$_2$CrO$_3$FeAs, does not appear to be superconducting, but the chromium...
ions are ordered antiferromagnetically while the iron atoms are non-magnetic. These compounds are a little unusual in that they appear to show none of the typical magnetic correlations associated with superconductivity in the iron layers that are found in other pnictides. We were therefore interested in investigating these compounds to see whether or not they were truly different from other iron pnictide families.

One of the major experimental problems was that it is difficult to synthesise pure samples. This is because although the materials are prepared by heating stoichiometric amounts (i.e. in the correct molar ratios) of the relevant oxides to very high temperatures (1000 K or more), vanadium, chromium and iron ions are the same size and so can act as a substitute for each other in the structure. This makes it difficult to establish the true chemical composition of the resulting material.

The chromium compound has proved particularly difficult to prepare with pure stoichiometry, since chromium always seems to find its way to the iron layers during the high-temperature preparation. Nevertheless, we synthesised almost pure samples of Sr₂CrO₃FeAs, and then analysed the structure using neutron powder diffraction which, in contrast to the analogous X-ray method, allows us to distinguish the positions of the chromium and iron ions in the crystal structure. The diffraction patterns were recorded at various temperatures using the high-flux powder diffractometer, D20, at the ILL.

The data unambiguously confirmed that some chromium always occupies the iron positions in the iron-arsenide layers, and so interferes with superconductivity and accompanying magnetic correlations that might be found in the version of the compound with pure iron layers.

We also took further powder diffraction patterns with polarised neutrons over a range of temperatures, using the DNS spectrometer at the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), to separate out the scattering peaks that are due to magnetic structure. We found that, below 36 K, there was long-range antiferromagnetic ordering of the chromium moments, which line up in one direction in one plane and in the opposite direction in the neighbouring layer in a checker-board arrangement.

Does magnetism have a role?
We then looked in more detail at the vanadium compound, which is the only family member that is superconducting. It had been speculated that the vanadium atoms might specifically play a part in the superconductivity. Furthermore, since it appears to be only the pure vanadium compound that is superconducting, and not the doped version as with other pnictides, it may be that the multivalent nature of vanadium allows the transfer of electrons to and from the iron layer, thus intrinsically doping it. The scandium compound (Sr₂ScO₃FeAs), which has a fixed valence of 3+, shows no superconductivity. We were interested to find out whether there was any magnetic ordering of the vanadium ions.

To investigate further, we aimed to control the composition of Sr₂VO₃FeAs by optimising the synthesis, in order to carry out combined X-ray and neutron powder scattering experiments. We made two versions: one that was the pure stoichiometric compound, and one with a small amount of excess vanadium substituting for some of the iron ions,
Sr$_2$VO$_3$Fe$_{0.93}$V$_{0.07}$As. As expected, the former showed a broad superconducting transition at 33 K; the latter showed no superconductivity.

Neutron powder-diffraction patterns at 300 K and 4 K were recorded with the high-resolution powder diffractometer SPODI at FRM II to determine the ratio of iron and vanadium in the iron layers. The data on the doped sample showed that 7% of the iron atoms had indeed been replaced by vanadium. The superconducting, undoped sample was shown to be almost stoichiometric. The data also showed additional peaks in the scattering pattern at 4 K, which might indicate additional, possibly helical, magnetic ordering of the vanadium ions, although this is still uncertain.

From these experiments, we conclude so far that very small amounts of impurity doping with chromium or vanadium into the iron-arsenide layer do poison the superconductivity in this pnictide family. It probably behaves similarly to the other pnictide families in that there is some magnetic ordering in the non-iron layers. The general absence of iron magnetism, and the possible self-doping effects in the vanadium compound, remain open questions for further study. It is possible that if we could prepare a stoichiometric chromium compound, it would be superconducting. Purer samples and better control of the stoichiometry will therefore be needed to understand the somewhat puzzling behaviour of the 21311 pnictides.

References

1. "Non-stoichiometry and the magnetic structure of Sr$_2$CrO$_3$FeAs", M. Tegel et al., EPL, 2010, 89, 37006.

Dirk Johrendt is Professor of inorganic solid state chemistry at Ludwig-Maximilians-Universität München, Germany.
The multiferroic compound, manganese tungstate, has an unusual low-temperature phase with a spiral magnetic structure that coexists with electrical polarisation. Such materials show promise as the basis of a new type of data storage using both magnetic and ferroelectric properties. Selected polarised neutron beams generated and analysed in three dimensions were used to investigate the possibility of controlling and remembering a magnetic state via an external electric field.

Recently, there has been a major resurgence of interest in multiferroic compounds – materials that simultaneously undergo more than one electrically or magnetically-driven phase transition. Of particular interest are those in which ferromagnetism (long-range ordering of atomic spins) and ferroelectricity (electric polarisation via ordering of atomic dipoles) exist together. The resulting magnetolectric coupling allows the electric polarisation to be changed by an external magnetic field, and the magnetic polarisation to be changed by an electric field. The latter effect could have huge potential in designing new types of memories for data storage, as well as applications in spintronic devices. However, very little is known about the reading and writing of such multiferroic states.

Indeed, very few multiferroics are known to science, and materials of real practical value have yet to be discovered. However, a new class of metallic oxide is being investigated in which polarisation develops when the magnetic spins are non-collinear – spirally ordered. Such exotic states tend to arise because of ‘frustration’ where there are competing interactions between neighbouring ions.

Manganese tungstate, MnWO₄, is one such compound. It has a zigzag arrangement of magnetic manganese ions and undergoes three magnetic phase changes on cooling to low temperatures:

- At 13.2 K, there is a transition to a collinear magnetic phase, AF₃, which is incommensurate, i.e. the periodic arrangement of spin directions does not coincide with the periodicity of the crystal lattice.
- At 12.3 K, an incommensurate but non-collinear spiral phase, AF₂, develops; it is the cycloidal ordering in which the direction of spins rotates along a perpendicular axis that simultaneously induces ferroelectric polarisation in an orthogonal direction. It is quite a small effect, being three orders of magnitude smaller than that of a standard ferroelectric material such as barium titanate, BaTiO₃.
- On further cooling to 7.0 K, the magnetic order becomes commensurate and collinear, and the ferroelectric polarisation disappears.

Magnetoelectric properties

We decided to study the effects of temperature and electric fields on the magnetic ordering in the AF₂ spiral phase, which would be the basis of any application in data-storage devices. Magnetic scattering with neutrons of precisely known polarisation detects changes in the alignment of magnetic moments in a material. Using the IN12 cold triple-axis spectrometer at the ILL, in combination with the Cryopad set-up, which allows the polarisation of the neutrons to be independently analysed in three-dimensions before and after scattering, we could identify the chiral component (associated with right or left-handedness) of the spins in the sample.

We were able to grow a fairly large single crystal for the experiments, and using an electric field, it was possible to achieve a ratio of the chiral components of more than 80% at the (-0.214, 0.5, 0.457) magnetic Bragg reflection. We carried out two sets of cycles of heating and cooling under electric fields with opposite polarisations, and took magnetic measurements of the AF₂ phase down to 6 K.

First, the crystal was cooled in a negative electric field of 875 volts/millimetre, which gave an almost perfectly aligned chiral arrangement of spins, designated A. On reheating to 15 K, above the AF₃ tran-
sition, and cooling again without the electric field, the A arrangement remains intact. Heating again and re-cooling, and then applying an electric field in the opposite direction, switches the alignment of spins to the opposite chirality, B. However, re-heating and cooling in zero field, this time switches the sample back to the A state. In other words, the magnetic moments on the manganese ions remember their original chiral alignment.

To start the second set of thermal cycles, we had to heat the sample to room temperature to erase the memory of A. We repeated all the cycles, but starting with the field in the opposite direction so that alignment B formed first. The same magnetoelectric memory effect was seen (though slightly weaker). We postulate that this memory effect is due to ‘pinning’ effects resulting from defects or distortions in the crystal structure. The memory and hysteresis behaviour of MnWO₄ appear to be complex and not entirely repeatable, as it seems to depend on how quickly the sample is cooling when it crosses the magnetic transition points.

Through the magnetic looking-glass
We then turned our attention to controlling the chiral arrangement by just varying the electric field, while keeping the temperature constant. The first hysteresis cycles of the chiral ratio versus the electric field were measured at 8.5 K under two different sets of initial conditions: in the first experiment, the sample was first cooled to the required experimental temperature in the presence of a negative voltage, and in the second, it was cooled from 15 K using a positive voltage.

In the first case, the chiral ratio remains unchanged right through the hysteresis cycle down to zero field and also when the voltage is reversed up to +1000 volts. This is probably due to the pinning mechanism mentioned earlier. Only when the positive voltage is increased much further does the chiral ratio significantly drop. However, cycling the voltage back through to its original negative field strength brings the chiral ratio rapidly back to its starting value.

The hysteresis cycle starting with a positive voltage is completely different: the initial chiral ratio is the same but of opposite sign. Lowering the voltage immediately reduces it, and it fully changes sign when the voltage is increased in the negative direction. When driving the negative voltage back again to zero, the reversed chiral ratio remains, and only partially recovers its initial state when the voltage is increased back to its original positive value.

We then repeated the hysteresis cycles at higher temperatures and found...
Towards a novel magnetic memory?

that at around the temperature of the transition from the AF2 to the AF3 spiral magnetic/ferroelectric phase, the same effects occurred but were much larger. However, all hysteresis curves are asymmetric, as before, indicating a preferred chiral arrangement. At 12.0 K, reversing the electric field induces a complete inversion of the chiral arrangement. This demonstrates that this kind of magneto-electric control could, indeed, be used as a basis for data storage.

These hysteresis experiments indicate that the magnetic and electric polarisation properties of manganese tungstate are quite complicated. Close to the transition temperature to the multiferroic phase, full magnetic reversal is possible but the hysteresis cycle remains very asymmetric. Well below the transition temperature, a full chiral inversion could not be obtained in our large crystal with the limited electric fields, because the chiral domains appear to be efficiently pinned. Even after heating the crystal above the first magnetic transition (AF3) temperature, it tends to remember the chiral arrangement enforced by the preceding field-cooling cycle.

Hysteresis curves of the chiral ratio as a function of external electric field at constant temperature; (a) to (d) were recorded after cooling in an electric field of 2000 volts; only the lower cycle in (a) was recorded after cooling in +2000 volts. Curves (e) to (l) were recorded after cooling in ~3500 volts.

Cryopad opened: the superconducting precession coils used to drive the incident and scattered neutron polarisation vectors.

Thomas Finger is a PhD student and Markus Braden is Professor at Cologne University, Germany.

References


Heavy-metal atoms, such as molybdenum, vanadium, manganese and iron, readily combine with oxygen and other atoms to form nano-sized clusters with a diverse range of structural and electronic characteristics. For example, if the cluster contains magnetic ions (i.e. with unpaired electron spins) and is well separated from other clusters by ligands (linking groups of atoms), magnetic interactions within a molecule are much stronger than those between molecules; the cluster behaves as a discrete ‘molecular magnet’.

Molecular magnets are of great interest because they often show unusual quantum behaviour at the mesoscopic scale. These mesoscopic spin states can be highly correlated and so can be used to realise ‘qubits’, the basic units of a quantum computer. These novel devices, which aim to exploit quantum principles to store and process unprecedented amounts of information, are now the focus of a great deal of research activity.

In some systems, nearest-neighbour ions may couple in a way that their electron spins tend to align antiferromagnetically (in opposing directions). If the three near-neighbours reside on an equilateral triangle, it becomes impossible to achieve simultaneous antiferromagnetic coupling for every possible pair of magnetic ions. This is known as magnetic ‘frustration’; it means that the electron spins can never settle down into an orderly arrangement with a minimum energy, or ground state – even at the lowest temperatures – since there are always at least two competing configurations possible. Such molecular magnets provide a useful microscopic model for investigating the subtleties of unusual magnetic configurations and behaviour at the quantum level.

A monster molecule
One of the largest frustrated molecular magnets ever synthesised is an iron polynuclear, abbreviated to [Mo\textsubscript{72}Fe\textsubscript{30}]. This 1000-atom cluster has a highly symmetrical structure containing 30 magnetic iron ions that sit at the vertices of an icosidodecahedron (a polyhedron with 20 triangular faces and 12 pentagonal faces). Antiferromagnetic coupling among the neighbouring iron ions occurs via magnetic exchange interactions transmitted through the surrounding framework of 72 molybdenum and oxygen atoms. The spin configuration represents the 3D molecular analogue of a well-known

**Unusual magnetism**

Frustration in a molecular magnet

Zhendong Fu

Experiments with polarised neutrons have uncovered the delicately balanced ground-state magnetic structure of a large metal cluster \([\text{Mo}\textsubscript{72}\text{Fe}\textsubscript{30}]\). The observed scattering pattern matches theoretical predictions.
Frustration in a molecular magnet

An equilateral spin triangle with antiferromagnetic interactions, demonstrating magnetic frustration.

Magnetic diffuse scattering at 1.5 K (red circles) and the theoretical simulation (blue line) based on the three-sublattice spin model.

similar planar model called a Kagome lattice, which is known to be highly frustrated.

The large number of atoms present in \([\text{Mo}_{72}\text{Fe}_{30}]\) means that it is difficult to analyse its magnetic structure. The simplest picture describes the 30 spins as divided into three sublattices of spins in the same plane and direction. We decided to investigate the magnetic structure directly using polarised neutrons to see if the model was a good fit.

We used the diffuse neutron scattering spectrometer, DNS, at the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), which is a cold neutron multi-detector time-of-flight spectrometer optimised to study exotic magnetic materials. It can analyse the changes in polarisation angles of the scattered polarised neutrons, and so separate out the magnetic scattering signals from nuclear scattering. We made measurements over a range of temperatures from 100 K to 1.5 K.

Below 20 K, we saw evidence for the presence of short-range antiferromagnetic spin correlations but no long-range magnetic ordering.

To determine the nature of the short-range magnetic ordering, we compared our experimental data with a computer simulation based on the three-sublattice spin configuration model, and could show that the magnetic diffuse scattering pattern matched well. Additional specific heat measurements also supported the model, so we deduce that the model offers a good approach to understanding the fascinating behaviour of this large and complex molecular magnet.

Zhendong Fu
is a postdoctoral researcher at JCNS, Forschungszentrum Jülich, Germany.

References
"An approach to the magnetic ground state of the molecular magnet \([\text{Mo}_{72}\text{Fe}_{30}]\)", Z-D Fu et al., New J. Phys., 2010, 12, 083044.
In the past 25 years, there has been an explosion of interest in the electronic and magnetic properties of mixed metal oxides and similar minerals, particularly those containing transition metals and/or rare earths. These heavy metal ions often have a variable valency; for example, iron ions within the crystal lattice can have either a 2+ or 3+ charge, and may be arranged such that there is geometric or magnetic frustration (whereby conflicting binding forces or magnetic coupling between ions prevent the configuration from settling into a unique minimum-energy state). As a result, these compounds can show a wide range of interesting magnetic and electric behaviours that are closely related to their crystal structures and exact composition.

The compound, lutetium ferrite, LuFe$_2$O$_4$, has attracted interest in recent years. The constituent iron ions show a 2+/3+ charge order and are also responsible for the magnetic properties of the material. They sit in bilayers consisting of triangular arrangements of iron and oxygen, which creates a highly frustrated configuration of both magnetic spins and charges. We used magnetic measurements and neutron scattering to map out for the first time the magnetic order and magnetisation of LuFe$_2$O$_4$ over a range of temperatures and magnetic fields. Figure shows the magnetic phase diagram obtained.

Below about 220 to 240 K, LuFe$_2$O$_4$ shows magnetic ordering, although it can also form ‘spin-glasses’ in which the magnetic moments are frozen in random directions due to frustration. Small changes in the composition, in particular the oxygen stoichiometry, which varies slightly from sample to sample, strongly affect what kind of magnetic state ultimately forms. We found that even samples from the same batch showed widely differing effects.

We carried out polarised neutron diffraction in zero magnetic field on DNS at the Forschungs-Neutronenquelle Heinz

**Unusual magnetism**

**Magnetism in randomly stacked layers: what’s in the flip of a spin?**

Joost de Groot and Manuel Angst

Lutetium ferrite, LuFe$_2$O$_4$, shows some fascinating magnetic behaviour. Its bilayered structure results in the formation of alternative magnetic spin structures with almost identical energies.
Magnetism in randomly stacked layers: what’s in the flip of a spin?

Maier-Leibnitz (FRM II) and non-polarised neutron diffraction in fields up to 2.5 tesla on D23 at the ILL, and the C5 spectrometer at the NRC Chalk River Laboratories in Chalk River, Canada, together with complementary soft X-ray diffraction at the Swiss Light Source (SLS) at the Paul Scherrer Institut in Switzerland. We focused our studies on samples with diffraction patterns showing the sharpest magnetic reflections.

**A metamagnetic transition**

The data showed that below 240 K, LuFe$_2$O$_4$ becomes antiferromagnetic. This is in contrast to previous suggestions that it was ferrimagnetic (i.e. with spins oppositely aligned but with a slight excess of magnetic moment in one direction) at this temperature. Applying an increasing magnetic field abruptly introduces additional reflections associated with a sudden magnetisation to the ferrimagnetic state. As the temperature is lowered, there is increased lagging in this magnetisation (hysteresis), which we interpret as a metamagnetic transition between the two spin structures.

These spin structures were assigned to the neutron data using a computer model that calculated all possible spin configurations of the iron spins for both the zero-field and high-field results. In the zero-field antiferromagnetic structure, the spins of each component layer of the bilayer were shown to be ferrimagnetically aligned, but with the net moments of the bilayers stacked antiferromagnetically. In the high-field ferrimagnetic version, all the spins in one bilayers were flipped, leading to twice as many spins in one direction as in the other, to give an overall magnetic moment.

The magnetic field–temperature phase diagram shows that at the magnetic transition temperature, the antiferromagnetic and ferrimagnetic structures have just about the same energies. Above 240 K, this ‘near degeneracy’ results in a random stacking of still ordered bilayers, as shown by diffuse neutron scattering. This phase competition is the hallmark of geometrical frustration and suggests LuFe$_2$O$_4$ as a model system of frustrated layered magnets.

---

**References**


Joost de Groot

is a PhD student in the Young Investigator Group, Complex Ordering Phenomena in Multifunctional Oxides, headed by Manuel Angst, PGI, Forschungszentrum Jülich, Germany.

---

The magnetic field–temperature phase diagram, which demonstrates a paramagnetic (PM) an antiferromagnetic (AFM) and a ferrimagnetic (fM) phase, extracted from various M(H) and M(T) curves. Arrows across the phase lines indicate for which measurement direction the transition is observed.

The spin structure in monoclinic C2/m cell of the AFM (a) and fM phase (b). Grey arrows indicate a net bilayer magnetisation. For better visibility, an additional iron layer is shown above the unit cell.

**JCNS Report 2009 - 2011**
Polymers

One of JCNS’s fields of expertise lies in the design, construction, and application of spin echo spectrometers, which offer unique insights into the dynamics of polymers.
No ends in sight

Ana Brás

The way in which polymers behave when molecular chains form rings is predicted to be markedly different from that of their linear analogues with free ends. Neutron spin echo (NSE), combined with small angle neutron scattering (SANS), has uncovered their dynamics for the first time.

Polymers, both natural and synthetic, are one of the most important classes of soft materials in everyday use. Their industrial processing and application depend upon understanding the behaviour of the constituent chains at the microscopic level. The complex motions of individual chains as they relax in response to forces due to surrounding molecules can be directly related to the all-important viscoelastic and flow properties of a polymer melt.

Two models have been developed to describe the chain dynamics: the Rouse model, which describes short polymer chains as beads linked by springs that diffuse via Brownian motion, and the reptation model for longer, entangled chains, which are considered to slither through a tube formed by neighbouring molecules.

Following the slithering (reptation process), relaxation occurs via the wriggling of the chain ends, which deconstruct the tube (contour length fluctuations), and the lateral movement of the surrounding chains (constraint release).

These models have been successfully used to predict the bulk properties of a wide variety of polymer structures including branched chains. However, in
ring structures, which have no chain ends, the mechanisms for relaxation are less clear. Indeed, flow experiments have indicated that ring polymers behave somewhat differently from their linear counterparts.

Ring polymers are thus of great interest in building up a complete picture of the relationship between the structure and dynamics of polymer molecules and the resulting bulk behaviour. They also have particular relevance for materials such as DNA, which can form rings, and thus have increasing technological potential.

So how does one polymer ring move in a melt of surrounding rings? So far, experimental exploration has been limited by the difficulty of making pure materials containing only rings and thus, free from contamination with linear chains, even a very small number of which can dramatically alter the physical properties of materials.

Pure rings
Recently, we were able to make considerable progress by preparing very pure samples of poly(ethylene oxide) (PEO) rings, and examining their intricate movements using NSE combined with SANS. Using the JNCS SANS diffractometer, KWS-2, we could characterise the structure and conformation of low molecular-weight rings, while quasi-elastic measurements obtained using the spin-echo spectrometer, JNSE, allowed us to follow the dynamics over timescales up to 100 nanoseconds.

The rings were too small to become entangled, but we could explore the fluctuations underlying diffusion associated with the Rouse model, and compare the results with those of linear polymers with the same molecular weight.

The SANS and NSE measurements, respectively, showed that the rings were much more compact, taking up only one-third of the volume occupied by the linear chains, and diffused considerably faster. This is in contradiction to the Rouse model, which predicts that the diffusion for both structures should be the same.

In collaboration with other research groups, we also measured the viscosity of both polymer melts and carried out molecular dynamics (MD) simulations to compare the theoretical description with the experimental results. The MD simulations supported the neutron data obtained, and also predicted that the viscosity of the rings would be only half that of the linear chains, which was in line with the viscosity measurements.

Our interpretation of the results is that the fast diffusion of the low molecular-weight rings is due to the much more compact conformation. Future plans are to extend the studies to higher molecular-weight rings allowing us to unravel relaxation mechanisms like reptation and constraint release in ring systems. We hope to be able to observe how the rings interpenetrate each other and so test one proposed picture of ring behaviour – the ‘lattice animal model’ – in which rings extrude long loop-like extensions that slide through a lattice of fixed obstacles to create animal-like shapes.

References
Polymer dynamics

Moving in confined spaces

Margarita Krutyeva

The motions of polymer molecules in bulk has been well studied. However, polymer dynamics in confined environments is less well understood. Neutron methods are now uncovering the complex behaviour of molecular chains in porous media, which will help in the rational design of new, commercially important materials and nanotechnologies.

Understanding the interactions of polymers confined in environments structured at the nano-scale is key to many applications exploiting the latest developments in nanotechnology. Membranes or sieves with nano-sized pores that selectively hold back particular macromolecules (or even viruses), while letting others through, have a wide range of potential applications in industry, domestic products and healthcare. Molecular sieves are also used as templates for nanofabrication; for example, linear polymer melts confined in nanoporous aluminium-oxide channels can form tailored one-dimensional structures – from nanotubes to nanorods – depending on how strongly they wet the pore walls.

Many of the properties of these systems depend on how the polymers infiltrate the pores and whether they stick to the pore surfaces, which in turn relates to their molecular size, structure and chemistry. Exploring the detailed movements of individual polymer chains within the pores offers an excellent probe of this behaviour. The Rouse and reptation models (p. 30) together provide a robust theoretical platform for describing the dynamics of short chains and longer, entangled chains over a range of timescales. Those models are already being successfully applied to pure polymer melts, as well as polymer composites containing nanoparticles (p. 35) where confinement effects become significant at high particle loadings.

The microscopic behaviour of polymers in a solid matrix has been less well studied, although some computer simulations have been carried out. To probe the applicability of the current theories, our research group decided to explore the effects of confinement on model polymers in a well-defined system – that is, with pores of known size and geometry – using neutron scattering.

Neutron methods for all timescales

A battery of neutron scattering techniques is now available that allows us to study different types of dynamical behaviour that characteristically occur over different timescales, from picoseconds to hundreds of nanoseconds. Time-of-flight (TOF) spec-
troscopy covers changes up to one-tenth of a nanosecond, while backscattering (BS) experiments measure dynamics over the nanosecond timescale. These techniques can be used to access motions over corresponding length-scales from one-tenth of a nanometre to a few nanometres.

With these methods, we aimed to explore the influence of the confinement on localised displacements (Rouse modes) within the macromolecules, and to check whether any interaction between the polymer and pore walls was visible. Neutron spin echo (NSE) measures processes taking place over longer times up to the microsecond scale, and can specifically look at motions concerned with entanglement, in particular the movements of the polymer chains along an effective tube constructed of neighbouring chains, as described by the reptation model.

We chose a pore system based on alumina (anodic aluminium oxide, AAO), which is fairly transparent to neutrons, thus allowing the polymer chains to be seen. The AAO material had well-defined parallel cylindrical pores of about 40 nanometres across, in which we could investigate the dynamics of a model polymer, polyethylene oxide (PEO). The hydrogenated PEO chains were introduced into AAO pores by spreading a thick liquid layer on the AAO surface, annealing the remaining layer to form a film, and then sealed.

In the first set of experiments employing the TOF spectrometer, TOFTOF, and the BS instrument, SPHERES, both at the neutron source Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), measurements were taken for two orientations parallel and perpendicular to the pores, and over a range of temperatures. The DNS spectrometer at FRM II was used to characterise the empty and filled porous matrix, revealing that the AAO was amorphous and the PEO partially crystallised. It was also possible to establish that the amount of the scattering from the PEO hydrogen atoms was much larger than the residual scattering from the nanoporous matrix.

These measurements allowed us to look at differences in molecular motions (associated with the PEO hydrogen atoms) flowing radially towards the pore walls, as well as in the direction along the pore axis. They showed that there was a slowing down of molecular motion close to the walls (perpendicular component), compared to what is seen in the bulk material. Calculations indicated that this was due to single, nanometre-sized layer of polymer molecules temporarily adhering to the wall sides. The data also showed that localised molecular fluctuations along the pore axis were unaffected by confinement. Nuclear magnetic resonance experiments had suggested that confinement narrows the effective tube size, as calculated from the reptation model, to produce a ‘corset’ effect that heavily restricts molecular movement to less than 1 nanometre. However, our measurements showed otherwise. Only tube diameters above 1.5 nanometres could be compatible with our experimental results. We can emphasise the absence of any effective tube with a diameter less than 1.5 nanometres.

Entanglement uncovered
To study the effects of the entanglement that appears on longer time/length scales, we then carried out NSE using the JCNS spectrometer, J-NSE at FRM II, on two PEO samples – one with polymer chains that were smaller than the width of the pores (weak confinement) and one in which they were larger (strong confinement). The samples were partially deuterated, and then contrast-matched to the
Moving in confined spaces

porous AAO whose scattering strength had been determined in a previous SANS experiment on KWS-1. In this way, the scattering due to the motions of the polymer chains could be highlighted.

The NSE experiments confirmed that for both short- and long-chain polymers, the chain dynamics were the same in the porous samples as in the bulk for short timescales, but slowed down over intermediate timescales in line with previous experiments, thus indicating some interaction with the pore walls. What happens to the level of entanglement, as observed in longer timescale measurements, is less clear. When the chain size is larger than the pore size, i.e. in the case of weak confinement, the entanglement seems to be reduced.

To look more closely at the effects of the pore walls on confinement, another researcher in our group investigated the behaviour of PEO and a hydrophobic, long-chain hydrocarbon, n-hexatriacontane (C36), in a more hydrophilic material – porous silicon – in which the pores were much larger than the molecules. We would expect PEO to be attracted to the silicon, while C36 would be less so. The NSE spectra were collected at different temperatures for different sample orientations so that molecular motions along the pore axis together with radial motions could again be analysed. The data revealed two sets of dynamics for both polymers – one where the molecules are immobile and stick to the pore walls, and one where they are fully mobile as they are in the bulk material. For C36, a molecular bilayer forms around the melting temperature, with the long chains lying parallel to the pore walls, but at increasingly higher temperatures, the layers gradually disintegrate. In the case of PEO, a single adsorbed layer forms, that is stable over the time measured by NSE.

These experiments demonstrate the effectiveness of neutron measurements in investigating the dynamics of polymers in confined environments. We are already extending our studies to other polymers and proteins to probe their adsorption, and the inter-chain friction and entanglement, in confining geometries.

Intermediate scattering function of PEO hydrogens (circles) in the AAO templates oriented at 135° allowing the measurement of the dynamics perpendicular to the pore axis at Q=1.4 Å⁻¹. The dashed red line presents a fit by a sum of 87% of the fast component (green solid line) attributed to the bulk and 13% slow component (dot black line) related to the surface layer.

References


Margarita Krutyeva is a postdoctoral researcher at JCNS, Forschungszentrum Jülich, Germany.
Inside polymer composites

Gerald J. Schneider

Neutron experiments, combined with theoretical models of polymer dynamics, are providing a powerful route to predicting the properties of advanced composite materials.

Materials consisting of polymers blended with nano-sized particles have exceptional properties that are finding use in an increasingly wide range of high-tech applications. They can confer mechanical strength or stiffness, and improved processability. For example, car tyres fabricated from particle-reinforced rubber have a lower rolling resistance that saves energy and increases longevity. Nano-structured composite materials also offer lightweight structural alternatives to metals such as those used in the aerospace industry: they account for 50% of the fuselage and wings of the new Boeing 787 Dreamliner. Lithium-ion batteries based on silica nanocomposites are promising candidates for use in electric vehicles.

It is well known that adding hard nanoparticles to a flexible polymer lowers its viscosity, making it stiffer. However, to formulate ideal materials for particular applications, their large-scale physical behaviour needs to be related to the detailed dynamics happening at the molecular level - for example, how the nanoparticles influence the movements of individual polymer chains, and how that relates to properties like stiffness. Physicists have, over the years, developed a theoretical microscopic approach to describing polymer dynamics. This encompasses the Rouse model (p. 30), which describes the random segmental motion of chains, and the reptation model (p. 30), which applies to long, entangled polymer molecules in which the chains are regarded as sliding, snake-like, through virtual tubes created by neighbouring chains. Disentanglement (relaxation) occurs, either when the chain escapes its current tube confinement via the movement of the chain ends, or through widening of the tube. In pure polymer melts, these models successfully predict viscoelastic properties.

How far can these models be applied to polymer nanocomposites? Attaining a full understanding of the molecular dynamics in nanocomposites has proved a challenge. Computer simulations, theoretical calculations and experiments addressing dynamical mechanical properties have been carried out, but the answers have often been ambivalent. Simulations have surprisingly indicated that, as the nanoparticles confine the movement of the chains, the degree of entanglement is reduced.

The effects of particles
To explore whether such ideas are correct requires the direct observation of the behaviour of both polymer molecules and nanoparticles. Small angle neutron scattering (SANS) and neutron spin echo (NSE) can provide exactly this information at the appropriate scale, i.e. in the range of a few nanometres and over several hundred nanoseconds. These neutron techniques have long been used to characterise the structure and dynamics of polymers, so we wanted to apply them to the characterisation of nanocomposites.
Nanocomposites

The reption model

Particles without the polymer

Polymer chains confined by particles

We carried out a series of experiments on a well-defined polymer–particle system that would allow us to separate out the various factors affecting its mechanical properties. The blend consisted of poly(ethylene-propylene) and silica nanoparticles that had been coated with short-chain hydrocarbons to render them hydrophobic. This ensured that there were no interfering, attractive polymer–particle interactions.

The first experiments performed on the KWS-2 instrument at the neutron source Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II) to examine the effects of various proportions of nanoparticles on the conformations of two polymer samples: one with short-chain molecules (PEP3k) that were much smaller than the particles, and one with long chains (PEP50k) that were about the same size. The components were each characterised separately. The PEP samples were prepared in both deuterated and hydrogenated forms to allow contrast variation (p8) such that they could be highlighted in the nanocomposite melt. Subsequent measurements with SANS indicated the coil sizes (8.33 nanometres in the case of PEP50k, and 1.93 nanometres for PEP3k).

The scattering intensity for the particles also had to be assessed in order to measure their size and to ‘contrast match’ them (p8) in the nanocomposite experiments, so that only the polymer coils would be seen. This was carried out in a mixture of normal and deuterated toluene with an average contrast that matched that of the particles in the nanocomposites. In this way, the direct scattering contribution from the particles can be measured and then removed from the final SANS data.

In fact, we discovered that it was impossible to achieve complete contrast matching, most likely because of the hydrocarbon shell surrounding the particle core, which created an additional contribution. In the SANS measurements of the sample blends, there was always a signal due to particle scattering, which was stronger at higher particle fractions. Nevertheless, this could be removed to give a corrected scattering contribution for the polymer component.

We found that for the short chains, their size was not affected by the particles, as predicted by the computer simulations. For the longer chains, the coil size was reduced in nanocomposite.

Polymers in motion

These results then enabled us to evaluate the polymer dynamics in the nanocomposite with NSE, which measures changes over the same length-scales as SANS. The experiments were carried out on IN15 at the ILL on blends of the longer-chain PEP with increasing amounts of nanoparticles, and they explored the range of relaxation mechanisms as expressed in terms of the Rouse and reptation models. We found that the shorter-timescale Rouse fluctuations were unaffected by the presence of particles even at high filler concentrations. However, in the case of the longer-period motions controlled by entanglement, the ‘tube’ considered to confine the movement of a single polymer chain (according to the reptation model), gradually narrows with increasing proportions of filler particles. At high concentrations of particles, they become the main obstacles to movement, while the polymer molecules themselves gradually disentangle, probably due to the squeezing effect generated by the immobile particles.

To predict technologically important bulk properties such as viscosity, we needed to link them to this new picture of reptation with a tube diameter modified by the additional confinement effects of the particles. Using NSE we could determine the tube diameter, and from the dynamical mechanical measurements, we could calculate the longest relaxation time, i.e. the reptation time. A comparison of the microscopic and macroscopic experiments confirmed that the confinement imposed by the nanoparticles causes the slowdown of the reptation time.

These results mean that we are now on the verge of being able to predict the properties of more complex nanocomposites (for example, where there are attractive interactions between polymer and filler) from the microscopic dynamics, as determined by neutron experiments.

References
Neutrons do not damage delicate structures such as those found in biological materials. JCNS instruments allow the probing of complex biomolecular structures, revealing their realistic behaviour in a watery environment similar to that found in living cells.
The life and times of polymer micelles

Lutz Willner

Polymers composed of hydrophilic and hydrophobic segments can dissolve in solution to form a variety of composite structures at the nanoscale level. A technique unique to neutrons, TR-SANS, is elucidating what drives their dynamic behaviour.

In solution, amphiphilic block copolymers readily self-organise into small aggregates called micelles. Their structure and behaviour depend on the relative hydrophilic/hydrophobic nature of both solvent and polymer components. In water, for example, the micelles form such that the insoluble hydrophobic components of each chain gather into a compact core. This is surrounded by the soluble hydrophilic part, which spreads out into the solvent to form a shell or corona. The self-organising process leads to a wide variety of fluid structures – from spheres, cylinders and vesicles to continuous polymeric networks – in which the individual polymer chains (unimers) are continually exchanging positions. Micelles are of great commercial interest, not only as drug-delivery vehicles, but also as components of complex fluids in foods, cleaning materials and other everyday products.

Understanding how micelles form and behave is therefore of great importance, in particular the rate at which the unimers are exchanged between micelles. When two micellar solutions are mixed, the exchange process randomises the distribution of the unimers in the micelles at a rate that decays with time until equilibrium is reached. Depending on the system, the time taken can vary between milliseconds and years (in which case, the system is effectively ‘frozen’). The accepted theory for the kinetics of exchange, developed in the late 1980s (by A. Halperin and S. Alexander), had proposed that it was the insertion and expulsion of unimers in the micellar structure that dominated the rate, rather than diffusion. It predicted that it was a simple first-order process characterised by a single exponent.

Testing the theory by measuring exchange rates in copolymer micelles presented a formidable challenge. However, during the past decade, we have been able to explore the kinetics and underlying influences, thanks to a technique we developed at JCNS, that of time-resolved small-angle neutron scattering (TR-SANS). These experiments, carried out on the KWS-1 and KWS-2 instruments at the neutron source Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), have provided important insights into the formation and dynamical behaviour of polymeric systems in solution.

Mixing and matching

The principle is to make two versions of micelles: one composed of fully deuterated polymers and one of non-deuterated polymers. The solvent used is partially deuterated so as to attain equal SANS intensities for both types of micelles. The micellar solutions are then mixed together, and SANS measurements are taken at regular time intervals. The rate of exchange of unimers between the two samples can be followed since the SANS intensity gradually decreases as the average excess fraction of either deuterated
or non-deuterated (proteated) chains in micelles decreases. Contrast matching (p. 8) and complete equilibration (equal distribution of labelled and unlabelled polymer chains inside the micelles) is achieved when the SANS intensity is minimal. The deuteration does not affect the kinetics, which can be clearly determined, and SANS provides the appropriate spatial resolution, which is monitored simultaneously.

The first experiments were carried out manually, but the method has since been automated using a stopped-flow machine, such that the mixing and the data collection are synchronised to allow measurements to be made with a time-resolution of just 50 milliseconds.

The first studies were carried out on a model system, poly(ethylene-alt-propylene)-poly(ethylene oxide) copolymer (PEP–PEO). This was dissolved in mixtures of water/dimethyl formamide (DMF), which is a selective solvent for PEO, and results in star-like micelles with a PEP core and PEO corona. The exchange kinetics in micelles consisting of these long-chain polymers are proportionately much slower than in micelles comprised of smaller detergent molecules. In water, due to the increased chain length and the high insolubility of the core polymer, the exchange is effectively frozen. However, by adding DMF and increasing the temperature, we are able to tune the kinetics such that they progress over a timescale accessible by SANS, i.e. from minutes to hours. We carried out the experiments over a range of temperatures between 40 and 80°C to determine the activation energy for the exchange.

Initial results carried out on an extended star-like copolymer, PEP1–PEO20 (the numbers indicate the relative molecular weights), were perplexing. The kinetics of equilibration did not follow the single exponential decay as expected, but a logarithmic rate. This suggested a broad distribution of equilibration times not predicted by the theory. The polymers were fairly pure with a narrow dispersion of chain lengths, and we thought that concerted interactions between the highly confined chains in the micelle could be the cause. However, later experiments, by another research group on a different polymer
The life and times of polymer micelles

system, indicated that the exchange kinetics were extremely sensitive to the chain lengths of the polymer, in particular in the hydrophobic component.

In our most recent experiments, we set out to explore further the effects of chain-length on the exchange kinetics. We used a series of polymers, each with a PEO component and a water-insoluble section consisting of a hydrocarbon chain with 18, 24 or 30 carbon atoms (C\textsubscript{18}-PEO5, C\textsubscript{24}-PEO5 and C\textsubscript{30}-PEO5). The micelles that form in water have large PEO coronas and a dense hydrocarbon cores. Each polymer was prepared so as to uniquely carry a mono-disperse core block in two versions, one with the PEO component proteated and one deuterated. These polymers allowed us to study the influence of the chain length of the cores directly – and thus the effect of polydispersity – on the equilibrium kinetics in block copolymer micelles. The technique described above was used to study the equilibrium kinetics.

Goldilocks polymers

We found that the C\textsubscript{18}-PEO5 micelles underwent unimer exchange too quickly to be measured, while the rate for the C\textsubscript{20}-PEO5 sample was much too slow. The rate for the C\textsubscript{24}-PEO5 copolymer was just right, and the decay in intensity could be measured over 100 seconds. The results showed that the exchange rate clearly followed a single exponential, and – taking into account the huge effect of hydrocarbon chain-length – confirmed the original theory.

In another, more complex experiment, we examined the effect of micellar morphology on the exchange kinetics using the block copolymer PEP1PEO1, which has short chains of equal length and forms ‘crew-cut’-type micelles with small coronas in water/DMF solutions. Their shape depends on the ratio of the solvent components: in water-rich solutions, the micelles are cylindrical but spontaneously transform into spheres in a solution containing 50% molar DMF. At intermediate DMF concentrations, this transition can also be induced irreversibly by heating for several hours. Thus, by dissolving the copolymer in a carefully chosen water/DMF mixture, we could follow the kinetics of mixing for both cylinders and spheres without changing the solvent ratio or the concentrations.

As in previous experiments, careful contrast-matching with the solvent was carried out. TR-SANS experiments on the two morphologies were performed on D11 at the ILL, and the results were compared with theoretical predictions that, this time, took into account variations in polymer-chain lengths. Unsurprisingly, the spherical micelles showed slightly faster exchange kinetics than the cylinders.

We hope to extend the research to other polymer combinations, in particular those that form transient networks rather than micelles, and use this neutron technique to relate the kinetics at the microscopic level to their bulk properties.

Lutz Willner

is a staff scientist at JCNS, Forschungszentrum Jülich, Germany.

References


Proteins are long-chain macromolecules that are responsible for much of the functional behaviour characterising the processes of life at the microscopic level. Each protein consists of a particular sequence of amino acids that folds up in a hierarchical way to present a complex structure with a specific three-dimensional shape and chemistry. A key molecular feature of many proteins is the ‘active site’ – a small region where a selected small molecule (substrate) binds, so as to induce a structural or chemical change that forms part of a biochemical pathway in a living cell.

Much of the early understanding of the function of proteins has come from the X-ray analysis of their crystal structures. This provides a good but unrealistically rigid 3D model of a particular protein and how it binds with its substrate. In living systems, proteins exist in solution and their overall conformations are likely to be somewhat different from those in the solid state, and furthermore, they are also free to move. Biologists have realised that to have an accurate understanding of protein function, they need to elucidate the detailed chain of movements within different parts of a protein as it interacts with a substrate under physiological conditions. Many proteins consist of molecular subunits, or ‘domains’, that appear to move in a cooperative way so that the substrate can ‘dock’ onto the active site.

Neutron scattering provides the ideal tool for probing the behaviour of proteins in their natural aqueous environment. The two techniques of small angle neutron scattering (SANS) and neutron spin echo (NSE) can respectively reveal their structures and dynamics over the relevant length- and time-scales (nanometres and nano-seconds). At JCNS, we have been conducting a pioneering research programme to explore the use of these techniques in studying functional protein dynamics. A decade or so ago, NSE could probe only the overall diffusion of proteins in solution; the neutron intensity and instruments available were not good enough to detect their internal movements. However, the past few years have seen important technical advances that have led to success. In particular, the instrument used for NSE at the ILL, IN15, which was developed and financed jointly by the ILL, Forschungszentrum Jülich and Helmholtz Zentrum Berlin (former Hahn-Meitner-Institut Berlin) has been upgraded; the new high-flux neutron source, FRM II in Garching, now provides improved opportunities for investigating biological macromolecules using SANS.

**Hinged proteins**

One of our first successes, reported in 2008, was the direct observation of the internal motions of a large protein whose 3D structure is well-known: alcohol dehydrogenase (ADH). This encompasses a family of enzymes containing zinc that interconvert alcohols and aldehydes, or
The natural rhythms of life

Ketones, with the aid of the cofactor, nicotinamide adenine dinucleotide (NAD+), and is responsible for alcohol detoxification. ADH can exist in a double (dimer) form, or as two dimers combined (tetramer). Each subunit (monomer) is dumb-bell-shaped with two globular domains connected by a 'hinge' that allows the structure to open up the cleft-like entrance to the active site.

We carried out a series of neutron experiments to isolate the movements of these domains.

Using instrument V4 at the Helmholtz Zentrum Berlin, SANS enabled us to establish the structure at the length-scale of the domains. A solution was prepared with a deuterated buffer in order to enhance the visibility of the protein to neutrons. The diffusional movement of the whole protein in solution was also identified via separate dynamic light scattering (DLS) experi-

ments. We could then use these results, combined with predictions from theory, to help pick out the collective motions of the domains in NSE experiments, carried out on IN15. Using a theoretical model that describes possible dynamical modes as though the protein is built of balls connected by elastic springs, we identified, for the first time, the dynamical mode related to the hinge action that changes the width of the cleft and allows the cofactor to enter or leave the site.

**Detailed neutron analysis**

We then followed up these results with similar, more detailed studies on phosphoglycerate kinase (PGK) – an enzyme involved in the glycolytic pathway (which processes sugars to release energy). It catalyzes the reversible conversion of 1,3-bisphosphoglycerate to 3-phosphoglycerate (3PG) by transferring a phosphoryl group during the synthesis of ATP (adenosine triphosphate) from ADP (adenosine diphosphate). PGK is composed of two widely separated domains also connected by a hinge region.

The binding sites for substrates ATP and 3PG are located with one on each domain so that they face each other. It had been suggested that after the binding of both substrates, the hinge action positions the substrates close together to enable catalytic activity. When the hinge then opens to about a nanometre, the ATP can emerge from the cleft.
Our goal was to characterise the exact motions of the domains, in terms of the amplitudes and directions of displacements, and their timing, both with and without the bound substrates. The SANS measurements, using KWS-1 at the FRM II, showed that the protein, when in solution, was more compact than in the crystalline state. There was a change in configuration when the substrates were bound, but the active site was not positioned closely enough to allow activity.

As before, we were able to extract the motions of the domains using NSE, by first subtracting the longer-lasting translational and rotational motions associated with diffusion, confirmed by DLS measurements. The observed excess dynamics could then be attributed to the large-scale internal dynamics of PGK. To analyse these further, we used our previous elastic network model to extract three modes of motion, the normal hinge motion, and torsional and rocking motions, to build up a picture of the overall configurational changes with and without substrates.

Our results showed that the intrinsic fluctuations of the protein are strong enough to cast it into a reactive configuration, whereby two or three critical amino acids in the domains come close together and initiate reactions involving the bound substrates. The thermally driven movements of the two domains take place on a timescale of 50 nanoseconds, which is fast enough to allow the necessary catalytic turnover of substrates.

These findings show that the internal dynamics of proteins are extremely important for their function. We hope that our neutron methodologies will continue to throw further light on the behaviour of these important biomolecules.

References

Drug delivery

Ruthenium-based nanovectors as anticancer drugs

Luigi Paduano

Ruthenium complexes, when combined with novel lipid analogues, offer a promising new route to safer, more effective cancer therapy. Small angle neutron scattering (SANS) is an essential tool in characterising these potentially valuable therapeutic candidates.

Platinum-containing compounds such as cisplatin, carboplatin and oxaliplatin are one of the most successful groups of drugs for the treatment of cancer. At least half of all cancer-related diseases are currently treated with cisplatin. However, this metal complex is highly toxic, causing severe and unpleasant side-effects for the patient, and thus limiting the effective dose that can be given. Pharmaceutical chemists have therefore been exploring the therapeutic potential of other, similar metals, and have identified complexes incorporating ruthenium as the most promising alternatives. A ruthenium complex called NAMI-A has been shown to be a highly active anticancer agent in vitro assays, and crucially has the advantage of being less toxic as it is more selective towards tumours, particularly metastases that tend to be difficult to treat. NAMI-A, along with two other candidate ruthenium molecules, KP1019 and RAPTA-C, has now completed Phase-I clinical trials.

There are still questions, however, about the effectiveness of the ruthenium complexes because of their apparent fast degradation under physiological conditions, i.e. in the presence of salts and active compounds such as enzymes typically found in living tissues. With this in mind, we decided to investigate potential molecular vectors that could transport the complexes to the tumour site while shielding them against environmental degradation over long circulation times, thus making the treatment more effective.

Designing the ideal vector

Lipid-based nanovectors make attractive candidates for transporting and delivering bioactive agents in the body. These chain-like molecules have long hydrophobic hydrocarbon tails and hydrophilic heads (amphiphiles) that readily self-assemble into ordered, nano-sized aggregates. They form well-defined layers or vesicles whose volume, shape and chemistry can be tuned to take up and release a controlled amount of bioactive material. Lipid-type molecules are also physiologically compatible and generally nontoxic, and can readily penetrate cell membranes, which are in turn constructed from phospholipids.

An example of the DoHuRu functional nucleolipid.
We designed a novel amphiphile using a nucleoside as a molecular scaffold – specifically uridine. The idea is that such a molecule would result in a non-toxic lipid-like structure that could readily recognise other bases such as the DNA bases of a cancer cell. It was chemically modified by attaching a methyl pyridine linker. This nitrogen-containing ring can readily grab onto a ruthenium atom to form a stable metal complex. The hydrophobic tail consisted of two chains, 18 carbon atoms long, attached at the 2' and 3' OH groups of the nucleoside. Hydrophilic polyethylene glycol (PEG) was inserted at the 5' position to act as a protective 'stealth' agent for the metal complex.

The resulting vector, called DOPURu, was dispersed in a solution mimicking physiological conditions, filtered and examined using a battery of techniques including SANS, which was carried out at the neutron research source Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II). SANS is typically able to access the length-scales associated with amphiphilic aggregates, and could confirm the presence of vesicles in solution. The experiments were a great success. The scattering showed that the vesicles consisted of stacks of lipid bilayers with a thickness of about 35 nanometres.

More recently, we extended the investigations to designing amphiphiles that were even more selectively nano-structured to mimic phospholipids and so reduce the toxicity of the drug in circulation, while enabling release in the target cells. We synthesised three nucleolipids, starting from either a uridine or thymidine central scaffold, to which one methyl pyridine arm (available to coordinate the ruthenium salt), one PEG arm and one or two hydrophobic oleic acid tails were attached. The two amphiphilic nucleosides based on thymidine, ToThy and HoThy, carried a single oleyl chain and different length PEG chains; the third amphiphile based on uridine, DoHu, had two tails (as is the case for phospholipids).

As before, we investigated their structures using SANS. The results confirmed that large layered vesicles formed under near-physiological conditions. The systems with the longer PEG chains had thicker layers, probably due to the PEG chains forming a compact hydrophilic shell in the presence of the surrounding water molecules. The ideal vesicle size for drug delivery is between 50 and 1000 nanometres. All the systems were within this range. In pure water, HoThy and DoHu also formed smaller cylindrical micelles.

We also carried out toxicity tests. Bio-screening on tumour and non-tumour cell lines revealed that the DoHu and HoThy aggregates did not show marked toxic effects, whereas ToThy showed a weak but significant cytotoxicity. Nevertheless, even a negative result helps us...
Ruthenium-based nanovectors as anticancer drugs

To understand the structure–activity relationships needed for tuning the design of biologically active nanosystems.

Towards biocompatibility
To improve the biocompatibility of DOPURu, as well as to increase the quantity of the metal complex that could be transported and delivered, we then investigated its aggregation behaviour in the presence of phospholipids, 1,2-dioleoyl-sn-glycero-3-phosphoethanolamine (DOPE) and 1,2-dioleoyl-sn-glycero-3-phosphocholine (DOPC). These form bilayers that aggregate into nanostructures, liposomes, that can take in the DOPURu and protect it from degradation until reaching the target cell, when a change in pH should result in delivery. SANS measurements indicated that mixtures of DOPURu with DOPE and DOPC did form a variety of stable nanostructures depending on the ruthenium complex/phospholipid ratio. To our surprise, however, cubic-shaped continuous networks, or cubosomes, also formed for a certain ratio. Cubosomes are already considered to be excellent candidates for drug delivery and other nano-designed systems such as foods, but previous formulations have not always been biocompatible.

In further pursuit of our aim to optimise our biomimetic vectors, we set about designing a system, based on the thymidine scaffold, that incorporated cholesterol as a cell delivery aid. Cholesterol is known to have a high affinity for phospholipids and thus would aid penetration through the membrane of a cancer cell. The system employed another naturally-occurring lipid, 1-palmitoyl-2-oleoyl-sn-glycero-3-phosphocholine (POPC), as a protective liposome, which had been shown to incorporate ToThyCholRu. The resulting nano-aggregates contained up to 15% molar concentration of the ruthenium complex, and were stable for several weeks, which is much longer than the bare NAMI-A, which lasts for only a few days in water.

SANS again was used to analyse the morphology of resulting aggregates, and showed that ToThyCholRu largely formed cylindrical micelles between 0.05 an 3.13 nanometres across, while the liposomes themselves had a multilamellar structure of about 4 nanometres but swelled up to 5 nanometres when ToThyCholRu was incorporated.

In vitro bioscreening of the ToThyCholRu/POPC liposomes also indicated that they were more effective in inhibiting the growth of human cancer cells. In particular, liposome systems are six times more effective than the corresponding non-amphiphilic ruthenium complex. We conclude that this approach of incorporating ruthenium complexes in biomimetic nano-systems offers enormous potential for drug therapy.

Luigi Paduano

is Professor of Physical Chemistry at the University of Naples Federico II, Centre for Colloid and Surface Science, Italy.

References
5. “A cholesterol-based nucleolipid-ruthenium complex stabilized by lipid aggregates for antineoplastic therapy”, Paduano et al., accepted in Bioconjugate Chemistry.
Inelastic neutron scattering experiments are uncovering how the subtle dynamics of lipid bilayers are affected by small molecules. The results could lead to a better understanding of the role of drug enhancers in medications.

Recently, we used inelastic neutron scattering to investigate in more detail how ethanol affects the behaviour of a lipid bilayer. By reflecting low-energy neutrons from supported model membranes, we were able to measure and analyse the small changes in energies associated with collective fluctuations in the membrane on a timescale of only 100 picoseconds. The samples were prepared as highly oriented stacks of 1,2-dimyristoyl-sn-glycero-3-phosphatidylcholine (DMPC-d54) bilayers with deuterated hydrocarbon tails to increase contrast, and spread out on silicon wafers. Stabilised layers with 5% ethanol were also prepared, to give a structure whereby each lipid molecule had an alcohol molecule attached. Using IN12, the cold-neutron triple-axis spectrometer operated by JCNS at the ILL, in conjunction with a vertically positioned analyser, we measured the inelastic
Moving membranes and molecular elevators

reflections at various energies and angles from the membrane, with and without the presence of ethanol. Computer simulations of molecular dynamics were used to interpret the results.

A new dynamic mode
As seen previously, the inelastic measurements revealed a collective acoustic ripple going across the pure lipid membrane. When ethanol was present, however, there was an additional low-energy mode that appeared to originate from a vertical movement traversing the membrane, which was associated with the lipid chains. We interpret this as coherent vertical waves passing up the tails. This scenario is supported by the diffraction data, which showed that the area occupied by each lipid molecule increased by about 4% in the presence of ethanol, so that the tails are less well packed.

These results may explain how ethanol and other small molecules such as aspirin act as drug enhancers. They may enable active transport across membranes, by creating a series of travelling voids and kinks in the tails that zig-zag up through the membrane and act as molecular elevators for drug molecules, thus allowing them to enter the cell more easily. We are now investigating the possibly similar role of aspirin to obtain a better understanding of the drug-delivery process into the cell at a molecular level.

Maikel Rheinstädter
is an Assistant Professor in the Department of Physics and Astronomy at McMaster University, ON, Canada. He is also affiliated with the Canadian Neutron Beam Centre at Chalk River, ON, Canada. Before moving to the US and Canada, Dr. Rheinstädter worked as a postdoctoral researcher with JCNS at the ILL in Grenoble.

References
JCNS instruments

Development programme

JCNS operates state-of-the-art instruments for neutron diffraction, reflectometry, small-angle scattering, and spectroscopy.
Aiming for the best

JCNS makes strenuous efforts to ensure that the scientific and technical quality of its instruments keeps in step with latest advances in modern neutron scattering techniques. JCNS instruments therefore undergo continuous upgrades with the addition of new technical options that enable them to remain among the best in their class.

The JCNS philosophy is to build the best instruments at the best neutron sources (FRM II, ILL and SNS, see page 64). We have thus created a unique suite of neutron instruments spanning both a wide length-scale, ranging from 0.05 nanometres (the hot neutron diffractometers) to 1 micrometre (the focusing SANS diffractometer), as well as a wide timescale, ranging from 1 picosecond (the thermal TOF spectrometer) to 1 microsecond (the neutron spin-echo, NSE, spectrometer at the SNS).

Dedicated neutron-delivery systems tailored for each of our instruments provide extremely high neutron fluxes, which not only significantly reduce the experimental time and sample size needed, but also enable studies of fast kinetic processes (using, for example, the stopped-flow technique).

One of the key elements of our development programme is the use of polarised neutrons. Here, we have already achieved remarkable progress, particularly with regard to the helium-3 (³He) neutron spin filters with steady polarisation, the superconducting precession coils and correction elements for the NSE spectrometers, and the grazing-incidence NSE technique.

JCNS is currently operating 10 neutron scattering instruments at the best neutron sources in the world – FRM II, ILL and SNS. Another four instruments – D23 and IN22 at the ILL, and BASIS and POWGEN at the SNS – are accessible to German users via a beam-time exchange arrangement with JCNS instruments at these sources. Two new instruments are under construction and will be installed at the new east neutron guide hall at FRM II.

BIODIFF – macromolecular diffractometer

In 2009, in cooperation with FRM II, we started the construction of a new diffractometer, BioDiff, for biological investigations that performs neutron diffraction studies on crystals with comparatively large unit cells. The unique feature of BioDiff, as compared to sister instruments at steady neutron sources such as LADI III at the ILL, is its possibility to adapt the neutron wavelength to the unit-cell size and the desired resolution. A similar effect can only be achieved by time-of-flight (TOF) instruments at spallation sources (for example, iBIX at J-PARC). BioDiff employs a large cylindrical image-plate detector covering roughly half of the total 4π solid angle. Additionally, a scintillation detector with a CCD readout is available for strongly scattering crystals. The construction of BioDiff was finished in 2011, and it has welcomed friendly users since March 2012.

Future developments will focus on the sample environment; for example, the integration of a cryostream that will allow measurements at temperatures down to 90 K.

HEIDI – single-crystal diffractometer

The single-crystal diffractometer, HEIDI, is installed at the hot-neutron source. The unit of three vertically-focusing monochromators provides a broad bandwidth of wavelengths and a high neutron flux, until now only available using D9 at the ILL. The optimisation of the beam geometry carried out during the long reactor shutdown in 2011 has allowed us to increase the flux for all wavelengths by about 20%. By using both the JCNS and TUM sample environments, it is possible to cover the temperature range between 2.2 and 1500 K.

Future developments of HEIDI will include the use of diamond crystals, which are well-known to be ideal monochromator...
Instruments

crystals for neutron scattering instruments. Recently, the technique for growing large, high quality diamond crystals (1.5 x 14 x 14 cubic millimetres) with growth direction (400) has been developed at the University of Augsburg. To raise the limits on the neutron wavelength band imposed by the small d400 spacing, the same crystals have been tested on HEIDI as transmitting (220) monochromators (the d220 spacings of diamond and copper are almost identical); a thin, 1.2-millimetre, single diamond crystal offers an impressive reflectivity gain of a factor of three to four, compared to a thick, 8-millimetre Cu(220) crystal at the same wavelength.

HEIDI, built and operated by RWTH, Aachen, joined JCNS in 2011.

POLI – polarised neutron diffractometer

The polarised neutron diffractometer, POLI, is currently operating on the monochromatic beam of HEIDI in beam-sharing mode. Neutron polarisation and polarisation analysis are carried out by 3He neutron spin filters. This separation of monochromatisation and polarisation is a unique arrangement for hot neutrons: in contrast to the use of Heussler monochromators, double-focusing monochromators combined with an optimised (tunable) polariser offer an intense polarised neutron flux at various energies (from hot to thermal neutrons) with variable resolution.

POLI is equipped with the zero-field spherical neutron polarimeter, CRYOPAD, that allows all components of the scattered polarisation vector to be determined, and gives access to the 16 independent correlation functions involved in most general nuclear and magnetic scattering processes. POLI has been available to users since 2010; however, it still shares the hot-neutron beam port and beam-time with HEIDI. At the end of 2011, the neutron beam port was modified, and new biological shielding was designed that will be manufactured during 2012, so that POLI will have its own neutron beam in 2013. This will in effect double the beam-time available for user experiments.

POLI, built and operated by RWTH, Aachen, joined JCNS in 2011.

MARIA – neutron reflectometer

A new-generation neutron reflectometer, MARIA, dedicated to studies of thin magnetic films, welcomed its first users at the end of 2011. To compensate for the weak scattering signal from small (thin) samples, the wavelength resolution and with that the neutron beam monochromatisation has been relaxed to 10%; a further, roughly three-fold gain in intensity comes from the elliptic neutron guide that focuses neutrons from the 17-centimetre-high neutron-beam port down to the 1-centimetre sample. By inserting absorbing horizontal plates in the elliptic neutron guide, MARIA can be instantly adapted for GISANS studies of laterally structured thin films.

Future developments include a Drabkin-type monochromator to tune the wavelength resolution in the range of 1 to 10% in a more or less continuous way (in contrast to the currently installed Fermi chopper with two fixed-wave resolution values of 1 and 3%) to extend possible applications of MARIA towards thicker films of up to 0.1 micrometre. MARIA is equipped with a position-sensitive detector (PSD) that is large for a neutron reflectometer (40 x 40 square centimetres) and allows for simultaneous data acquisition of the specular and off-specular signal or of a wide Q-range in GISANS measurements, thus leading to significantly shorter experimental times.
The JCNS instrument development programme

Further improvements require producing a large hybrid (potassium–rubidium) SEOP cell, about 14 centimetres across, made of a special glass, GE180. For this purpose, a special oven allowing in-situ glass welding will be installed and used in the Forschungszentrum Jülich glass workshop. A gas-filling station with precise pressure gauges has been constructed and is now being commissioned. Last but not least, the $^3$He recovery system, which allows us to sustain and to clean this valuable gas, has been put into operation.

KWS-1, KWS-2 and KWS-3 SANS diffractometers

Using the same technology, we have developed a compact polarisation analyser for biological structure studies with the SANS diffractometer, KWS-1. The absence of a magnetic field means that the distance between it and the $^3$He cell can be very short, thus increasing the potential Q-range up to 1 Å$^{-1}$. Capitalising on this advantage, we can separate coherent and incoherent contributions in the scattered signal, and increase the signal-to-noise ratio by two orders of magnitude, thus allowing the architecture of complex biological molecules to be determined in a single experiment.

KWS-1 is currently being prepared for polarised neutron experiments: a polarising cavity consisting of three V-shaped transmission supermirrors (from Swiss-Neutronics), combined with an RF adiabatic spin-flipper, will provide an average polarisation of 93% at 0.5 nanometres, and will be installed in front of the collimation base to suppress the diffuse spin-flip scattering contribution at the detector. The polariser will be installed on a revolving device to allow rapid switching between polarised and non-polarised modes, and will start in autumn 2012.

An extremely high flux (up to $2 \times 10^8$ neutrons per second per square centimetre) supplied by the cold neutron source and the newly designed neutron guide sys-
The JCNS instruments

KWS-2

This will extend the high Q limit by an order of magnitude, and overlap with the Q ranges of KWS-1 or KWS-2.

NSE spectrometers

New correction elements for the NSE spectrometer, J-NSE, have been developed and tested. These are modifications of Pythagoras coils initially developed at the ILL for the very high currents required for the strong magnetic field of the superconducting precession coils. This has not only allowed for the extension of maximal spin-echo time to 200 nanoseconds but has also practically doubled the intensity due to the replacement of copper with aluminium as the coil material. Future plans include the use of the full field integral for long wavelengths, replacing the middle set of correction coils by larger coils that are better adapted to the beam diameter in the second arm, and to increase the flux at long wavelengths by modifying the polarising optics. This capitalises on new developments in supermirror coatings and allows us to cover the whole usable wavelength band, from 0.5 to 1.7 nanometres, with a single polariser.

The SNS-NSE spectrometer at the Oak Ridge National Laboratory (ORNL) in the US became functional in November 2009, and the first user-friendly programme started three and a half years after the signing of the agreement between Forschungszentrum Jülich and ORNL. Further improvements of the NSE software and hardware continued until the instru-

JCNS Report 2009 - 2011
The JCNS instrument development programme

The JCNS instrument development programme was fully operational in 2011. The instrument improved tremendously in terms of data quality and reliability, and 81 days of operation time will be delivered to scientific experiments in the first user-cycle of 2012. Thanks to the new correction elements, the maximum resolution of this instrument is 450 nanoseconds; at the moment, it is only the lack of higher neutron flux at longer wavelengths that is preventing the attainment of higher resolutions beyond 450 nanoseconds. However, with the neutron source being further optimised to 1.4 megawatts, more neutrons will be available and the resolution will increase. The next generation of correction coils should allow for the further increase in the maximum resolution, up to 1000 nanoseconds.

IN12 cold neutron triple-axis spectrometer
The cold neutron triple-axis spectrometer, IN12, at the ILL is dedicated to elastic and inelastic studies of low-energy structural and magnetic excitations, and has proven its value over the years. At present, IN12 is moving to a new end position. A new guide with an adapted coating, and a horizontally-focusing and vertically-widened end part has been installed; the double focusing monochromator will lead to a substantial gain in flux of about one order in magnitude and an extension of the wavelength range down to 0.15 nanometres. This move to a new position has been used as an opportunity to upgrade and refurbish the primary spectrometer.

SPHERES high-resolution spectrometer
The high-resolution spectrometer SPHERES is a third-generation backscattering instrument with focusing optics and a phase-space transform chopper – a versatile spectrometer for investigating atomic and molecular dynamics on a GHz scale. Since the start of full user-operation in 2007, instrument performance has been continuously improved. The signal-to-noise ratio has been improved from 330:1 to 1700:1 by decreasing the fast-neutron background and filling the entire instrument with argon to avoid air-scattering in the secondary spectrometer. This value, as well as other principal figures of merit (spectral flux, resolution, dynamic range), qualifies SPHERES as one of the best in its class.

Future plans include the implementation of six diffraction detectors installed in the horizontal scattering plane along the large analyser panels, and covering scattering angles from about 30° to 120°. This will allow structural and dynamical information to be obtained simultaneously. A further gain in flux will be achieved by a more efficient phase-space transform chopper, which is currently under development.

TOPAS thermal time-of-flight spectrometer with polarisation analysis
The thermal time-of-flight spectrometer with polarisation analysis, TOPAS, will be built in the east neutron guide hall. The high neutron flux over a wide energy range
with a modest divergence will be delivered by a combination of an elliptic neutron guide and a set of Fermi choppers. The wide-angle $^3$He neutron spin filter will allow for XYZ neutron polarisation analysis over a wide solid angle subtended by a large array of PSDs. We developed an improved approach based on the ILL instrument, PASTIS, with mu-metal sheets producing an arbitrarily directed magnetic field that is homogeneous over a large area, and with a reduced blind area resulting from the support material of the Helmholtz coils that are usually used as the magnetic-field holding system. The present design has a nearly 45° window in the vertical direction, providing polarisation analysis for the full height of the detector. All components of TOPAS are now at the engineering design or manufacturing stage, with the exception of the PSD, for which the concept has to be changed due to the $^3$He shortage crisis. The spectrometer vessel with a volume of 70 cubic metres can be pumped to below $10^{-5}$ millibars within several hours. To avoid pumping the whole vessel when the sample environment is changed, the equipment has been designed so that a separate inner tank, which contains a 300-litre volume around the sample environment, can be pumped within minutes.

POWTEX time-of-flight diffractometer
Another instrument that will be built in the east neutron guide hall is the TOF diffractometer, POWTEX, which will fulfil the needs of solid-state chemists, geologists and materials scientists who rely on powder and texture diffraction. The instrument design combines a new concept of two double-elliptic neutron guides with the pulse chopper placed at the common focal point of one square centimetre. This optimises the time resolution and efficiently reduces the background from the reactor source. The second guide focuses neutrons onto a small sample spot of the same size as the pulse-chopper window; a uniform Gaussian shape for the intensity and divergence distributions at the sample will be achieved due to the guide’s octagonal cross-section.

The PSD detector will cover a huge solid angle, 10 steradians, which avoids reorienting the sample in texture measurements. As an alternative to the $^3$He detectors, wavelength-shifting-fibre detectors (designed at Forschungszentrum Jülich and a boron-10 solid-state detector (from CDT GmbH) are being developed: prototypes of both are in their final design stage. The use of polarised neutrons (without polarisation analysis) is planned as a future upgrade.

Sample environments
The JCNS suite of sample environments largely defines the experimental possibilities of our instruments. Besides the wide choice of cryostats and ovens allowing for studies in the temperature range from 650 K down to 20 mK, each of our instruments is equipped with a dedicated sample environment with features answering instrument specifics. Just to name a few: a compensated asymmetric cryomagnet is necessary for the polarisation analysis with $^3$He neutron spin filters at MARIa and DNS; an extremely high neutron flux available at KWS-1 and KWS-2 allows us to use stopped-flow apparatus for fast kinetic measurements; and non-magnetic pressure cells are being developed for the biological studies at the NSE spectrometers.
# List of instruments

In the period from 2009 to 2011, JCNS operated a total of 14 neutron scattering instruments at three different sources. Two additional instruments are currently under construction.

- Research Reactor FRM II (Forschungs-Neutronenquelle Heinz Maier-Leibnitz) in Garching, Germany
- High Flux Reactor at the Institut Laue-Langevin in Grenoble, France
- Spallation Neutron Source at the Oak Ridge National Laboratory, Oak Ridge, USA

## Diffraction

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>BIODIFF</td>
<td>Single crystal diffractometer for biological macromolecules (collaboration with Technical University Munich)</td>
</tr>
<tr>
<td>D23</td>
<td>Thermal neutron two-axis diffractometer for single-crystals (CEA instrument, access for German users through JCNS)</td>
</tr>
<tr>
<td>HEIDI</td>
<td>Hot single crystal diffractometer (operated by RWTH Aachen)</td>
</tr>
<tr>
<td>POLI</td>
<td>POLarisation Investigator using HEIDI monochromator (operated by RWTH Aachen)</td>
</tr>
<tr>
<td>POWGEN</td>
<td>Powder diffractometer (SNS instrument, access for German users through JCNS)</td>
</tr>
<tr>
<td>POWTEX</td>
<td>POWder and TEXture – High intensity time-of-flight diffractometer (under construction)</td>
</tr>
</tbody>
</table>

## Reflectometry

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>MARIA</td>
<td>Magnetic Reflectometer with high Incident Angle</td>
</tr>
</tbody>
</table>

## Small Angle Scattering

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>KWS-1</td>
<td>Small Angle Neutron Scattering diffractometer</td>
</tr>
<tr>
<td>KWS-2</td>
<td>Small Angle Neutron Scattering diffractometer for soft matter and biology</td>
</tr>
<tr>
<td>KWS-3</td>
<td>Ultra Small Angle Neutron Scattering diffractometer</td>
</tr>
</tbody>
</table>

## Spectroscopy

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>BASIS</td>
<td>Backscattering spectrometer (SNS instrument, access for German users through JCNS)</td>
</tr>
<tr>
<td>DNS</td>
<td>Diffuse Neutron Scattering spectrometer with polarisation analysis</td>
</tr>
<tr>
<td>IN12</td>
<td>Cold neutron three-axis spectrometer with polarisation analysis</td>
</tr>
<tr>
<td>IN22</td>
<td>Thermal neutron three-axis spectrometer with polarisation analysis (CEA instrument, access for German users through JCNS)</td>
</tr>
<tr>
<td>J-NSE</td>
<td>Jülich Neutron Spin Echo spectrometer</td>
</tr>
<tr>
<td>SPHERES</td>
<td>Backscattering SPectrometer for High Energy RESolution</td>
</tr>
<tr>
<td>SNS-NSE</td>
<td>Spallation Neutron Source - Neutron Spin Echo spectrometer</td>
</tr>
<tr>
<td>TOPAS</td>
<td>Time-Of-flight with Polarisation Analysis Spectrometer (under construction)</td>
</tr>
</tbody>
</table>
# Status of instruments

<table>
<thead>
<tr>
<th>Instrument</th>
<th>2009</th>
<th>2010</th>
<th>2011</th>
</tr>
</thead>
<tbody>
<tr>
<td>BIODIFF</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DNS</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HEIDI*</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>J-NSE</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>KWS-1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>KWS-2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>KWS-3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MARIA</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>POLI*</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>POWTEX</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SPHERES</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TOPAS</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D23**</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>IN12</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>IN22**</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>BASIS**</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SNS-NSE</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>POWGEN**</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* beam time allocation managed by TUM
** JCNS has guaranteed access to German users but does not operate the instruments

- Green: Commissioning phase
- Orange: Test period after a long break
- Blue: Regular operation
- Yellow: Under construction

---

* JCNS Report 2009 - 2011
List of sample environments available at FRM II

JCNS offers sample environments for a wide spectrum of applications. The offer is continuously updated to meet the demanding requirements of the users. The table below shows the range of equipment available at the FRM II outstation.

<table>
<thead>
<tr>
<th>Sample environment</th>
<th>Range</th>
<th>DNS</th>
<th>HEBI</th>
<th>JNSE</th>
<th>KWS 1</th>
<th>KWS 2</th>
<th>KWS 3</th>
<th>MARIA</th>
<th>POL-HEIDI</th>
<th>SPHERES</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cryostats</td>
<td>0.02 - 350 K</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>11</td>
</tr>
<tr>
<td>Cryofurnace</td>
<td>3 - 650 K</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>4</td>
</tr>
<tr>
<td>Electromagnet</td>
<td>up to 1.2 T</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>2</td>
</tr>
<tr>
<td>Pressure cells</td>
<td>up to 5 kbar</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>2</td>
</tr>
<tr>
<td>Cryomagnet</td>
<td>2 - 300 K, up to 5 T</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>1</td>
</tr>
<tr>
<td>Biofurnace</td>
<td>250 - 400 K</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>1</td>
</tr>
<tr>
<td>Small furnace</td>
<td>278 - 450 K</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>1</td>
</tr>
<tr>
<td>Peltier furnace</td>
<td>253 - 400 K</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>1</td>
</tr>
<tr>
<td>Rheometers</td>
<td></td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>1</td>
</tr>
<tr>
<td>Stopped flow cell</td>
<td>time resolution 50 ms</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>1</td>
</tr>
<tr>
<td>Humidity chamber</td>
<td>5 - 95%, 283 - 343 K</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>•</td>
<td>1</td>
</tr>
</tbody>
</table>
JCNS user programme

Access to state-of-the-art instruments

Scientists from universities and industry all over the world are welcome to apply for beam at JCNS.
The JCNS User Programme

JCNS state-of-the-art instruments are offered to the neutron scattering community worldwide.

Researchers from anywhere in the world wishing to carry out neutron scattering experiments are welcome to apply for beam-time at JCNS. The procedure is a simple one: researchers submit a project proposal electronically via the JCNS Online User Office. Our staff scientists are on hand to discuss the project, and to advise in detail on the preparation and submission of the proposal.

The review process then takes place around six weeks after the proposal deadline; members of the JCNS Review Panel decide on the allocation of beam-time in terms of the proposals’ scientific merit and the beam-time available. It is important to note that proposal rounds take place twice per year. Once a proposal has been accepted, the user is then invited to discuss the planning of the experiment with the designated local contact.

Financial Support
Two funding streams enable JCNS to offer its first-class instruments to external users, along with associated expenses:

- For users from German academic institutes, funding for neutron scattering experiments is available from the German Federal Ministry for Education and Research (BMBF).
- Users from other EU Member states or Associated States can apply for support through the NMI3 (Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy) scheme, which aims to promote the use of neutron scattering in Europe. Funding is managed via the TransNational Access programme. To be eligible, the home institutes of the group leader and the majority of the group must be based outside the country hosting the facility. During the period 2009-2011, a total of 56 user visits to JCNS to conduct experiments over 197 beam days were supported through this highly successful scheme.

The Soft Matter Community
A limited number of beam days is also available through two important European projects – SoftComp and ESMI:

- SoftComp was initially established as an EU Network of Excellence covering the field of soft matter composites. The present SoftComp Consortium has its own online proposal submission system and review panel. Beam-time and the accompanying financial support are confined to SoftComp members.
- The European Soft Matter Infrastructure (ESMI) is an ongoing EU infrastructure project due to run for a period of 48 months, and devoted to the promotion of soft matter research in Europe. Financial support and access to first-class instrumentation in all relevant experimental techniques is thus made available to the whole European soft matter community. Within this scheme, JCNS offers access to its neutron scattering instruments via the TransNational Access mechanism.

Financial support for all these schemes covers a maximum of two members per experimental team, and includes travel, subsistence and accommodation costs for the full period of the visit to JCNS.

The JCNS User Office is available to help users with the preparation and the submission of proposals, along with experiment planning, including logistics support in Garching.

Please feel free to contact the JCNS User Office by email at: neutron@fz-juelich.de.

Last but not least, companies can also benefit hugely from a cooperation with JCNS in terms of assistance with scientific investigations. From 2009-11, a number of experiments driven by problems in industry were carried out using JCNS instruments.

Companies can reserve beam time on JCNS instruments by contacting Dr. Rainer Bruchhaus (r.bruchhaus@fz-juelich.de).
NMI3 visits at JCNS

Distribution of NMI3 beam days to external visitors at JCNS in the years 2009–2011.
During the past three years, the JCNS neutron facility, FRM II, and its instruments were taken off-line for a year to undergo maintenance works and upgrades. Nevertheless, large numbers of users from across the world were able to access JCNS facilities over the remaining operational period, and take advantage of the improved facility.

1. FRM II reactor operation
During the period 2009-2011, the JCNS research reactor that provides neutron beams, FRM II, operated for 237, 219 and 61 days in 2009, 2010 and 2011, respectively. The low number of days in operation in 2011 is due to the long technical break from October 2010 to October 2011. Operations were back to normal by the end of 2011. During the long break, some instruments were upgraded, and the last cycle in 2011 was used mainly to check their improved performance before making them available to external users.
2. Beam days available at FRM II instruments

From 2009 to 2011, there were 997 beam days available in 219 sessions, for external experiments on JCNS instruments at FRM II. The pie chart shows the distribution over the available instruments and scientific areas. It should be noted that some instruments were undergoing commissioning during 2009 (KWS-1, KWS-3 and MARIA), and also 2010 (KWS-2, KWS-3 and also 2011 MARIA), and so were not available for user experiments. Moreover, all the instruments except J-NSE and SPHERES underwent some upgrading during the long reactor break, and therefore were only partially open to external research programmes during the single reactor cycle in 2011. Their distribution across the scientific areas shows that soft condensed matter, condensed matter physics and magnetism investigations cover about two-thirds of the total beam days provided. The ratio between the beam days used for external and internal research is 1.92 overall.

Number of experimental sessions vs. scientific areas FRM II

- Soft Condensed Matter 46%
- Condensed Matter Physics 16%
- Biology 13%
- Chemistry 10%
- Magnetism 10%
- Industrial Application Geo Sciences 1%
- Material Science 2%
- Crystallography 0%
3. Number of proposals received on all JCNS instruments available

The number of proposals received in 2009, 2010 and 2011 is 142, 211 and 119, respectively, for JCNS instruments at all facilities – FRM II, and the ILL and SNS outstations. The graph shows their distribution across both scientific areas and instruments. It should be noted that the number of proposals received in 2011 constituted only one proposal round due to the FRM II long break. This number is very encouraging when compared to the number of proposals received in the two previous years with two proposal rounds each. The number of proposals received for the FRM II, ILL and SNS instruments is 396, 32 and 44, respectively.
4. Requested/allocated beam days on all proposals received

Proposals received during the period 2009–2011 (via two proposal rounds in 2009 and 2010, and only one in 2011) for all JCNS instruments at the three facilities (FRM II, the ILL and SNS) requested 875, 1195 and 664 beam days, respectively. The total number of allocated days is 500, 578 and 273, in the years 2009, 2010 and 2011, respectively. The graph shows the number of requested and allocated days for all JCNS instruments. There are a number of overload factors of 1.75, 2.07 and 2.43 for 2009, 2010 and 2011, respectively, with an average overload factor of 2.02 for the combined three years. The low number of beam days allocated in 2011 is not surprising because of the high number of experiments that were approved in 2010 but could not be measured before the FRM II long break.
5. Home countries of the principal investigators of experiments carried out between 2009–2011

This pie chart shows the home country of the principal investigator for all the external research proposals approved and performed using FRM II instruments. About half of the principal investigators are German; however, JCNS also attracts successful proposals from a number of other countries, including France and the United States.

6. Home countries of visitors who carried out experiments between 2009–2011

Between 2009 and 2011, JCNS hosted 396 visitors performing experiments with the FRM II instruments over 997 beam days (see pie chart 1). Germany, France, the United States, the United Kingdom, Japan and Italy were each the source of more than 10 visitors. In total, JCNS instruments were used by visitors from 23 countries worldwide.
The Future of neutron scattering

European Spallation Source (ESS)

The European Spallation Source will be the most powerful neutron source in the world. JCNS works on the design and development of neutron instruments which make ideal use of the possibilities which ESS has to offer.
Contributing to the neutron source of the future

The European Spallation Source (ESS) is set to become the next generation European research facility built in Lund, Sweden. When it becomes operational in 2019, initially with seven instruments, it will be the most powerful neutron source in the world.

It is planned that the facility, complete with a total of 22 instruments, will be fully operational by 2025. Currently, 17 European countries are involved in this project, which has been given priority status on the roadmap of the European Strategy Forum on Research Infrastructures (ESFRI).

Forschungszentrum Jülich currently coordinates the German activities in the framework of the Design-Update-Phase of the ESS. The Federal Ministry of Education and Research is supporting this project, which runs from 2010 to 2013, with funding of €15 million. This secures Germany's position as a major partner in the ESS. Altogether seven German research centres participate in this joint project, which is managed by Prof. Dieter Richter (Jülich/JCNS) and Dr. Wolfgang Schroeder (Jülich/FS). Furthermore, Prof. Sebastian Schmidt, member of the Jülich Board of Directors, is the Coordinator of the all-German project and member of the international ESS Steering Committee. Scientists from Jülich are also members of the Scientific and Technical Advisory Committees.

Since mid-2011, activities undertaken by Jülich for the ESS have been bundled together in a newly-established ESS com-
European Spallation Source (ESS)

petence centre headed by Dr. Andreas Wischnewski. This centre comprises all work on innovative instrumentation concepts in JCNS, on the target station in the Central Institute for Technology (ZAT), and on concepts for detector electronics in the Central Institute for Electronics (ZEL).

Within JCNS, the ESS-Instrumentation department has recently been established. In addition to the department head, there are four staff (postdoctoral researchers) whose positions are funded up to 100% by the joint project. Work in the ESS-Instrumentation department is focused on the design and development of neutron instruments which are ideally adapted to the characteristics of the ESS so that the given pulse structure (repetition rate 14 Hz, pulse length 2.8 ms) is exploited in the most efficient way.

At present, concepts are being put forward for a range of instruments, including a high resolution spin-echo-spectrometer (NSE), a time-of-flight instrument (TOF), a powder diffractometer, a small-angle-neutron-scattering instrument (SANS) and a reflectometer. Furthermore, polarisation elements based on 3He are being developed. Beyond that, within the joint project, members of JCNS head a total of four separate work groups; their involvement extends altogether to seven different groups dealing with instrumentation concepts and critical instrument components.

The work done in Jülich for the ESS project is based on and strongly benefits from the decades of experience accrued with respect to both the design and construction of components for spallation sources and the respective neutron instrumentation.

Drawing of the Jülich layout of the high resolution spin-echo spectrometer that has recently been brought into operation at the spallation source SNS in Oak Ridge. Experience gained there is proving a beneficial guide in our efforts to develop an optimized spin-echo spectrometer design for the European Spallation Source, ESS in Lund.
Outreach activities

Education and dissemination in neutron science

Each year, JCNS organises a number of highly-regarded educational courses, workshops, and conferences.
The Jülich Centre for Neutron Science (JCNS) is extremely active, not only in stimulating interactions between neutron scattering specialists and users, which then lead to new technical and scientific advances, but also in promoting neutron scattering techniques and applications to the wider scientific community, including students. We sponsor and organise a wide-ranging programme of events aimed at the dissemination of the advantages and current status of neutron scattering methods, and their crucial role in cutting-edge research. We also provide an educational programme for students and new users, which is much in demand. The events are listed in Tables on page 75.

The JCNS educational programme

JCNS is proud to contribute to the education of the future generation of scientists. Our main educational event is the JCNS Neutron Scattering Laboratory Course, which we run annually. The course consists of a week of lectures at Forschungszentrum Jülich on topics relevant to neutron scattering, plus a week of experimental work at the JCNS laboratory at the research reactor FRM II (Forschungs-Neutronenquelle Heinz Maier-Leibnitz), on 11 instruments that are made available for an entire five day period. Students are divided into groups of five, and each group carries out five different experiments, one per day, covering all important aspects of neutron scattering. The experimental work includes special set-ups to improve the understanding of the instruments, and also model experiments relevant to real-life applications.

We are grateful for the support of the Technische Universität München (TUM), the RWTH Aachen University, LMU Munich, Technische Universität (TU) Darmstadt and the University of Göttingen, which kindly provide teaching staff and the use of five neutron scattering instruments (HEIDI, PUMA, SPODI, RESEDA and TOFTOF).

The 2009, 2010 and 2011 courses were attended by 53, 58 and 54 students respectively. Generally, far more students apply than are accepted because places are limited by the number of instruments available; indeed, about half of all applications are rejected. The cost of a JCNS Laboratory Course ranges from about 34 k€ (2009) to about 38.5 k€ (2010); all students are offered lodging and subsistence, and travel costs are granted to those students from non-German universities. Students’ feedback is always very positive – for both the theoretical and experimental parts.

The JCNS Laboratory Courses are made possible thanks to the financial support provided by the NM3 EU project, the SoftComp Consortium and the FeMAS EU project. Detailed and updated information on past and present JCNS Laboratory Courses is available at: www.fz-juelich.de/jcns/LabCourse/

JCNS has played a major role in the organization of recent IFF Spring Schools, both in 2010 at the school, entitled “Electronic Oxides - Correlation Phenomena, Exotic Phases and Novel Functionalities” and in 2011 at the one dealing with “Macromolecular Systems in Soft and Living Matter”. During these events, JCNS’s expertise on neutron scattering as well as on magnetism and soft condensed matter was made available to the groups of 197 and 186 students attending the respective schools, from around 30 different countries. The IFF Spring Schools aim to provide students with a solid introduction to the basic physics of the different disciplines with special reference to the interdisciplinary aspects involved, while presenting more advanced approaches to the core topics.

JCNS also runs schools that focus on special topics. In April 2010, the JCNS School on Data Analysis for QENS took place. This specialist school on quasi-elastic and inelastic neutron scattering, organised by Dr Joachim Wuttke, was devoted to users of time-of-flight and back-scattering techniques, and attracted up to 15 students – thus indicating the great interest in the topic. The two-day school consisted of a theoretical introduction to the basic concepts, and an introduction to the data-reduction and analysis of experimental data by means of SLAW and FRIDA software. The productive and encouraging atmosphere experienced by the students demonstrated the success of the school.

The JCNS dissemination programme

Every year, JCNS organises workshops on ‘hot topics’ in neutron scattering, as well as on neutron instrumentation.

“Trends and Perspectives”

One workshop series, “Trends and Perspectives in Neutron Scattering”, takes place every year in the autumn; in the
years 2009–2011, three workshops were devoted to soft matter, magnetism and instrumentation, respectively. There is always great scientific interest in these workshops, as demonstrated by the large number of highly respected scientists who participate. The 2009, 2010 and 2011 workshops were respectively attended by 65, 77 and 85 participants from all over the world.

The 2009 workshop, “Trends and Perspectives in Neutron Scattering on Soft Matter”, dealt with the structural and dynamical problems associated with soft matter materials such as polymers and proteins. An outstanding list of 21 invited and 17 selected speakers contributed to the success of the four-day workshop. The next workshop, “Trends and Perspectives in Neutron Scattering for Soft Matter and Biophysics”, will take place again in Tutzing, Germany on 8–11 October 2012. The financial contributions provided by the SoftComp Consortium and the Donostia International Physics Centre, San Sebastian, Spain, are gratefully acknowledged.

The 2010 workshop, “Trends and Perspectives in Neutron Scattering: Magnetism and Correlated Electron Systems”, included sessions on pnictides, multiferroics, quantum magnetism, magneto-calorics, frustrated magnets, heavy fermions, novel instrumentation and sample preparation. A total of 26 invited and 26 high-level scientific talks stimulated an intense and rewarding discussion amongst participants. The support of partners from industry (EADS Astrium, Oerlikon Leybold Vacuum, Pfeiffer Vacuum and SwissNeutronics) is greatly appreciated.
The 2011 workshop, “Trends and Perspectives in Neutron Instrumentation: from Continuous to Spallation Sources”, was devoted to the development of the neutron scattering instrumentation to be installed at the next-generation neutron sources, i.e. spallation neutron sources. The six invited and 57 selected talks focused on the most efficient way to use the available knowledge for the design and construction of neutron scattering instrumentation at spallation sources.

All three JCNS workshops enjoyed lively, constructive poster and discussion sessions, facilitated by the informal working atmosphere, the attractive venues and the recreational activities organised for participants in the pleasant Bavarian surroundings.

Detailed and updated information on the past and present JCNS Workshops is available at:

www.fz-juelich.de/jcns/Conferences/Workshops/

**Novel neutron techniques and their applications**

JCNS also runs workshops that promote the dissemination of information about novel neutron techniques and their applications. During 27-29 September 2009, a workshop on modelling and data-analysis related to off-specular reflectometry and grazing-incidence scattering was organised. More than 40 participants from Europe and the US attended, with many representatives coming from the major neutron and X-ray facilities. Many experimental examples in soft matter and in magnetism were presented, as well as the software for data analysis used. Lively discussions between participants resulted in an agreement to create a repository for the existing computer programs.

In connection with the inauguration ceremony of the JCNS Neutron Spin Echo Spectrometer at the Spallation Neutron Source operated by the Oak Ridge National Laboratory in the US – the first spin echo instrument ever built at a spallation neutron source – JCNS organised the “Neutron Spin Echo Spectroscopy 2009” workshop from 4-5 November 2009. Around 80 participants met to discuss the current status and trends in scientific fields driven by neutron spin echo investigations. As a result of increasing interest, JCNS also organised a two-day workshop on 12-13 July 2010, on the “Modern Trends in Production and Applications of Polarised 3He”. A lively and fruitful discussion stimulated by the 29 invited and selected talks, took place among the 50 or so participants, on the following topics: polarisation of $^3$He, $^3$He containers, magnetic holding systems, $^3$He spin manipulation and applications.

The international conference, “Neutrons for Global Energy Solutions”, was jointly organised with other major neutron facilities worldwide on 26-29 September 2010, with the aim of identifying potential contributions that neutron scattering could make to solving energy problems.
This event has strategically been very important for the neutron scattering community, which was able to discuss with the energy research community how neutrons could be applied to energy research. About 80 participants attended the event, which included round-table discussions with funding agencies and industrial representatives. It was agreed that neutron scattering has potentially a significant role to play in this vitally important area.

Last but not least, JCNS’ scientific horizons are much wider than those just represented in our workshops and conferences, and it is worth mentioning that JCNS provided sponsorship and scientific support to large national and international conferences, such as SNI 2010, Berlin, and ECNS 2011, Prague, and topical workshops in JCNS scientific fields of interest.

<table>
<thead>
<tr>
<th>Table 1: JCNS educational courses</th>
</tr>
</thead>
<tbody>
<tr>
<td>07-18 September 2009</td>
</tr>
<tr>
<td>08-19 March 2010</td>
</tr>
<tr>
<td>28-29 April 2010</td>
</tr>
<tr>
<td>06-17 September 2010</td>
</tr>
<tr>
<td>05-16 September 2011</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 2: JCNS workshops and conferences</th>
</tr>
</thead>
<tbody>
<tr>
<td>27-29 September 2009</td>
</tr>
<tr>
<td>05-08 October 2009</td>
</tr>
<tr>
<td>11-13 July 2010</td>
</tr>
<tr>
<td>26-29 September 2010</td>
</tr>
<tr>
<td>04-08 October 2010</td>
</tr>
<tr>
<td>04-07 October 2011</td>
</tr>
</tbody>
</table>
## FRM II

<table>
<thead>
<tr>
<th>Name</th>
<th>Position</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dr. Marie-Sousai Appavou</td>
<td>2nd Instrument scientist KWS-1</td>
</tr>
<tr>
<td>Dr. Earl Babcock</td>
<td>Staff scientist</td>
</tr>
<tr>
<td>Dr. Zhenyu Di</td>
<td>2nd Instrument scientist KWS-1</td>
</tr>
<tr>
<td>Dr. Artem Feoktystov</td>
<td>2nd Instrument scientist KWS-1</td>
</tr>
<tr>
<td>Dr. Henrich Frielinghaus</td>
<td>1st Instrument scientist KWS-1</td>
</tr>
<tr>
<td>Dr. Zhendong Fu</td>
<td>2nd Instrument scientist KWS-3</td>
</tr>
<tr>
<td>Dr. Olaf Holderer</td>
<td>1st Instrument scientist J-NSE</td>
</tr>
<tr>
<td>Dr. Andreas Houben</td>
<td>1st Instrument scientist POWTEX</td>
</tr>
<tr>
<td>Dr. Vladimir Hutau</td>
<td>1st Instrument scientist POLI</td>
</tr>
<tr>
<td>Dr. Alexander Ioffe</td>
<td>Head of the JCNS outstation</td>
</tr>
<tr>
<td>Dr. Denis Korolkov</td>
<td>2nd Instrument scientist MARIA</td>
</tr>
<tr>
<td>Dr. Stefan Mattau</td>
<td>1st Instrument scientist MARIA</td>
</tr>
<tr>
<td>Ms. Franziska Michel</td>
<td>Secretary</td>
</tr>
<tr>
<td>Dr. Martin Meven</td>
<td>1st Instrument scientist HEIDI</td>
</tr>
<tr>
<td>Dr. Harikrishnan Sasidharan Nair</td>
<td>Staff scientist DNS</td>
</tr>
<tr>
<td>Dr. Kirill Nemkovskiy</td>
<td>2nd Instrument scientist TOPAS</td>
</tr>
<tr>
<td>Dr. Vitaliy Pipich</td>
<td>1st Instrument scientist KWS-3</td>
</tr>
<tr>
<td>Dr. Sabine Pütter</td>
<td>Staff scientist</td>
</tr>
<tr>
<td>Dr. Aurel Radulescu</td>
<td>1st Instrument scientist KWS-2</td>
</tr>
<tr>
<td>Mr. Harald Schneider</td>
<td>Sample environment responsible</td>
</tr>
<tr>
<td>Dr. Gerald Johannes Schneider</td>
<td>2nd Instrument scientist SPHERES</td>
</tr>
<tr>
<td>Dr. Tobias Schrader</td>
<td>1st Instrument scientist BIODIFF</td>
</tr>
<tr>
<td>Dr. Yixi Su</td>
<td>1st Instrument scientist DNS</td>
</tr>
<tr>
<td>Dr. Noemi Kinga Szekely</td>
<td>2nd Instrument scientist KWS-2</td>
</tr>
<tr>
<td>Dr. Jörg Voigt</td>
<td>1st Instrument scientist TOPAS</td>
</tr>
<tr>
<td>Dr. Joachim Wuttke</td>
<td>Head of the JCNS Scientific Computing</td>
</tr>
<tr>
<td>Dr. Michaela Zamponi</td>
<td>1st Instrument scientist SPHERES</td>
</tr>
</tbody>
</table>

## ILL

<table>
<thead>
<tr>
<th>Name</th>
<th>Position</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dr. Haifeng Li</td>
<td>Polarisation analysis IN12</td>
</tr>
<tr>
<td>Dr. Karin Schmalz</td>
<td>1st Instrument scientist IN12 Head of the JCNS outstation</td>
</tr>
<tr>
<td>Dr. Wolfgang Schmidt</td>
<td>2nd Instrument scientist IN22 and D23, UFO-project (IN12)</td>
</tr>
</tbody>
</table>

## SNS

<table>
<thead>
<tr>
<th>Name</th>
<th>Position</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dr. Michael Ohl</td>
<td>1st Instrument scientist SNS-NSE Head of the JCNS outstation</td>
</tr>
<tr>
<td>Dr. Nikolas Arend</td>
<td>2nd Instrument scientist, SNS-NSE</td>
</tr>
<tr>
<td>Dr. Delphine J. Gout</td>
<td>Staff Scientist</td>
</tr>
<tr>
<td>Dr. Niina Jalarvo</td>
<td>2nd Instrument scientist BASIS</td>
</tr>
<tr>
<td>Ms. Sabine McNulty</td>
<td>Secretary</td>
</tr>
<tr>
<td>Dr. Melissa Sharp</td>
<td>2nd Instrument scientist, SNS-NSE</td>
</tr>
<tr>
<td>Dr. Laura Stingaciu</td>
<td>2nd Instrument scientist, SNS-NSE</td>
</tr>
</tbody>
</table>
What are neutrons, how are they produced and why are they indispensable for research? How does society benefit from neutron research and what are Forschungszentrum Jülich’s contributions? These are just some of the issues regarding the key technology of neutron scattering explored in the ten-minute video on the Jülich Centre for Neutron Science (JCNS), found on the DVD (page opposite). The film was produced in 2011 and is available in both German and English.

If the DVD is missing, please contact us or watch the video on our website: www.fz-juelich.de/jcns/.