

# Pressing the Neutron Guide Hall East to Completion

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**Don't forget to  
submit your proposal!  
Next deadline:  
January 25<sup>th</sup>, 2013**





## *The collaboration at the FRM II entered a new era*

Selecting applications for experiments purely on their scientific merit guarantees the high quality of the usage of the FRM II. This proposal selection relies on a strict peer review procedure and follows internationally established standards. Right from the beginning of user operation in 2005 the FRM II has organised such kind of review panels selecting the submitted proposals twice a year. A similar procedure was applied at the neutron centres in Jülich and Geesthacht until their shutdown in 2006 and 2010, respectively.

After the transfer of several instruments to Garching had been accomplished, the tradition of the review panel of JCNS was continued with great success. However, this led to an increased work load of the user office at the FRM II, as it had to organise two independent review sessions.

With the ratification of the collaboration agreement of the Technische Universität München with the institutes of the Helmholtz centres in Jülich, Geesthacht and Berlin in 2011, the goal was to establish a common review of all proposals submitted to the FRM II. In September 2012, the referees met for the first time for a joint selection of proposals, organised in six review panels dealing with applications in the area of biology, imaging analysis and nuclear and particle physics, magnetism and spectroscopy, material science, soft matter and structure research. Each of these review panels is lead by a chair person and supported by a secretary. 56 experts of international renown from 13 different European countries had to evaluate the proposals during a two-day meeting.

With this new organisation we aim to facilitate the extremely valuable engagement of our referees and ensure a fair, but sometimes difficult selection process versus our user community. We cordially invite our users to submit further proposals by January 25<sup>th</sup>, 2013, which will be evaluated in March by our next common review panel meeting.

*Flavio Carsughi*  
User Officer at the FRM II



## Upgrade of the Neutron Imaging Beam Line ANTARES

### Neutron Imaging

Neutron Imaging is based on the detection of the transmission of a neutron beam through an object. In contrast to X-ray imaging the mechanisms of interaction of neutrons and X-rays with matter are extremely different from each other. This leads to higher contrast for several materials (e.g. water, oil, etc.) and higher penetration for many metals compared to X-rays, making neutron imaging the method of choice for many special industrial and physical applications. Currently resolutions as good as 25  $\mu\text{m}$  can be achieved for both radiography (2D) and tomography (3D).

### The story of ANTARES

The imaging beam line ANTARES at FRM II was one of the first beam lines at the neutron source to come in operation in 2004 and since then had been one of the most intense imaging facilities in the world. Many successful experiments had been performed at ANTARES in various fields of scientific research and industrial application, which have profited largely from the high flux and good collimation. However, due to the construction of a new

neutron guide hall in the east of the FRM II reactor building the beam port SR4b at which ANTARES was located had to be cleared for the installation of a neutron guide to deliver cold neutrons into this new hall. Therefore, the old ANTARES beam line was dismantled and almost completely rebuilt at the neighbouring beam port SR4a. Taking this redesign of the beam line as an opportunity to improve the concept of the ANTARES beam line resulted in a major upgrade of the neutron imaging facility at FRM II now providing even better performance and higher flexibility.

### The new beam line concept

An improved concept was developed for the new ANTARES beam line. A key design feature was to provide – among new possibilities – identical beam conditions as on the old ANTARES so users can directly compare new measurements with old ones.

The beam is now accessible over the entire length of the instrument as shown in fig. 1. The first shielding block connected to the biological shielding houses a massive instrument shutter and a collimator drum which allows the selection of six different collimators with pinhole sizes between 2 mm and 70 mm. Furthermore, three chambers were installed along the beam path separated from each other by massive shielding walls to reduce the fast neutron and gamma background.

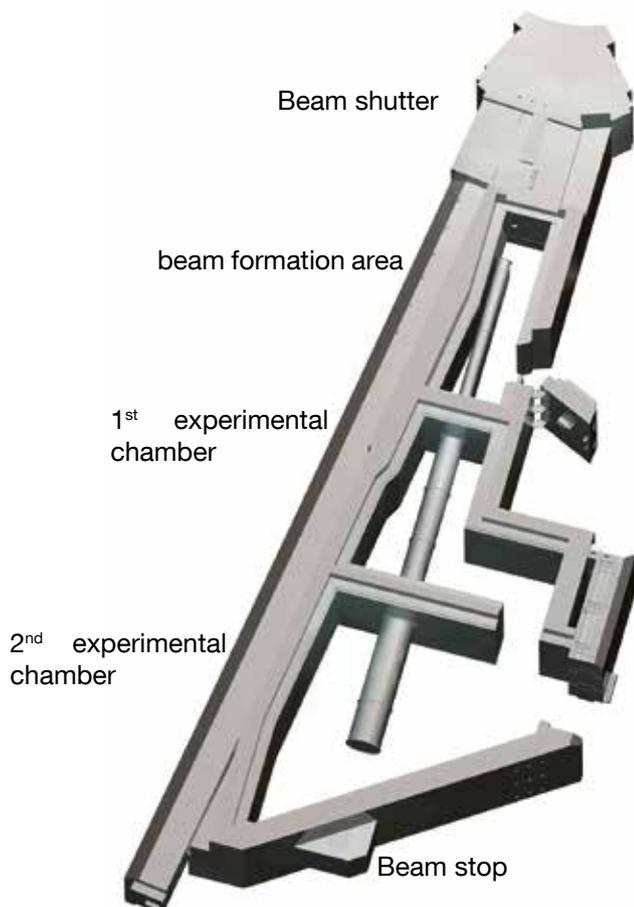


Fig. 1: The new design of ANTARES.



Fig. 2: ANTARES during reconstruction.

The first chamber in beam direction offers the possibility to flexibly install all kinds of beam forming devices. In the beginning it will host a fast shutter to reduce activation of the sample, a filter selector wheel with different crystals to shape the spectrum and reduce the fast neutron and gamma background. Furthermore, a double crystal monochromator and/ or a velocity selector will be installed as well as neutron optical periscopes to avoid direct sight to the beam tube. Devices provided by the user can also be mounted in this chamber.

Following the beam formation area, will be the first experimental chamber, where experiments with high flux on smaller samples can be performed. The maximum beam size in this area will be approx. 20 x 20 cm providing a flux as high as  $1.6 \cdot 10^9$  n/cm<sup>2</sup>/s for the largest pinhole, which renders this position ideal for stroboscopic or fast imaging of moving processes. Moreover, using smaller pinholes the collimation ratio L/D can be increased from 100 over 200, 400, 800 and 1600 even up to approx. 3000. Due to the small beam size in this chamber and the fact that the beam dump is located further downstream in the next chamber the background for high resolution or monochromatic imaging on small samples is significantly lower than at the old ANTARES beam line. The roof was elevated in a small area to accommodate the standard FRM II cryostats which do not fit into the last chamber.

The third and last chamber provides, among others, conditions identical to the old ANTARES with a maximum beam diameter of up to 35 x 35 cm and a massive sample manipulation stage to handle even large and heavy samples. A separate detector with a variable field of view of up to 40 x 40 cm is installed at this sample position.

Both experimental chambers offer a rail system installed in the floor which provides flexible and accurate positioning of all devices like flight tubes, sample manipulation stages, detectors, etc. along the beam direction.

A new, reusable shielding material based on iron powder, boron and paraffin oil cast into steel containers was developed which provides a better shielding of fast neutrons and gammas while having the same density as heavy concrete. This allowed us to decrease the thickness of the walls and therefore to increase the interior size of the beam line without exceeding the floor weight limit. The new ANTARES beam line now offers ample



Fig. 3: Philipp Schmakat is placing a sample on the new stage for small samples in the first experimental chamber.

space for even complex sample environment (e.g. power supplies, cryostats or gas handling systems for fuel cells) to be installed at both of the sample positions.

## Applications

The new features render the new ANTARES facility one of the most flexible and powerful imaging beam lines worldwide. We offer a variety of different detectors with different fields-of-view which provide a resolution of up to 25  $\mu$ m and time resolution for repetitive processes in the  $\mu$ s range.

The field of applications at ANTARES is vast and covers, among others, archaeology, geology, biology, fuel cells, construction materials, stroboscopic imaging of fast processes but also fundamental research on magnetism or the structure of superconducting vortices.

Only two years after the dismantling of the old ANTARES beam line was started, the new instrument is now near completion. We expect to be back in operation between spring and summer 2013 for standard experiments and you are very welcome to submit proposals for new experiments through the FRM II User Office online system.

*Michael Schulz, FRM II*



## 5 Tesla Actively Shielded Split Pair Magnet for SANS

A new magnet is now available for experiments using small scattering angles like SANS machines or reflectometers. The horizontal field can be applied perpendicular or parallel to the neutron beam with a maximum field of 5 tesla in the symmetric mode. For polarized neutron experiments requiring a guide field the magnet can be operated in an asymmetrical mode with a centre field up to 2.5 tesla.

The 80 mm vertical room temperature bore accepts the cryostat with a sample of up to 25 mm in diameter. The two conically shaped horizontal bores accept scattered neutrons from any point of the sample up to an angle of  $15^\circ$  with respect to the axis of the bore.

A dedicated pair of coils compensate the stray field to a magnitude of  $50 \mu\text{T}$  (earth field) in a distance of 1.2 m from the centre of the magnet as shown in the diagram (fig. 2). Hence, the influence on experiments at neighbouring instruments can be neglected.



Fig. 1: SANS-magnet with the conical room temperature bores visible (image with courtesy provided by Oxford Instruments).

Furthermore, no liquid helium is required: The completely dry cooling system provides convenient and reliable operation of the magnet.

*Herbert Weiß, FRM II*

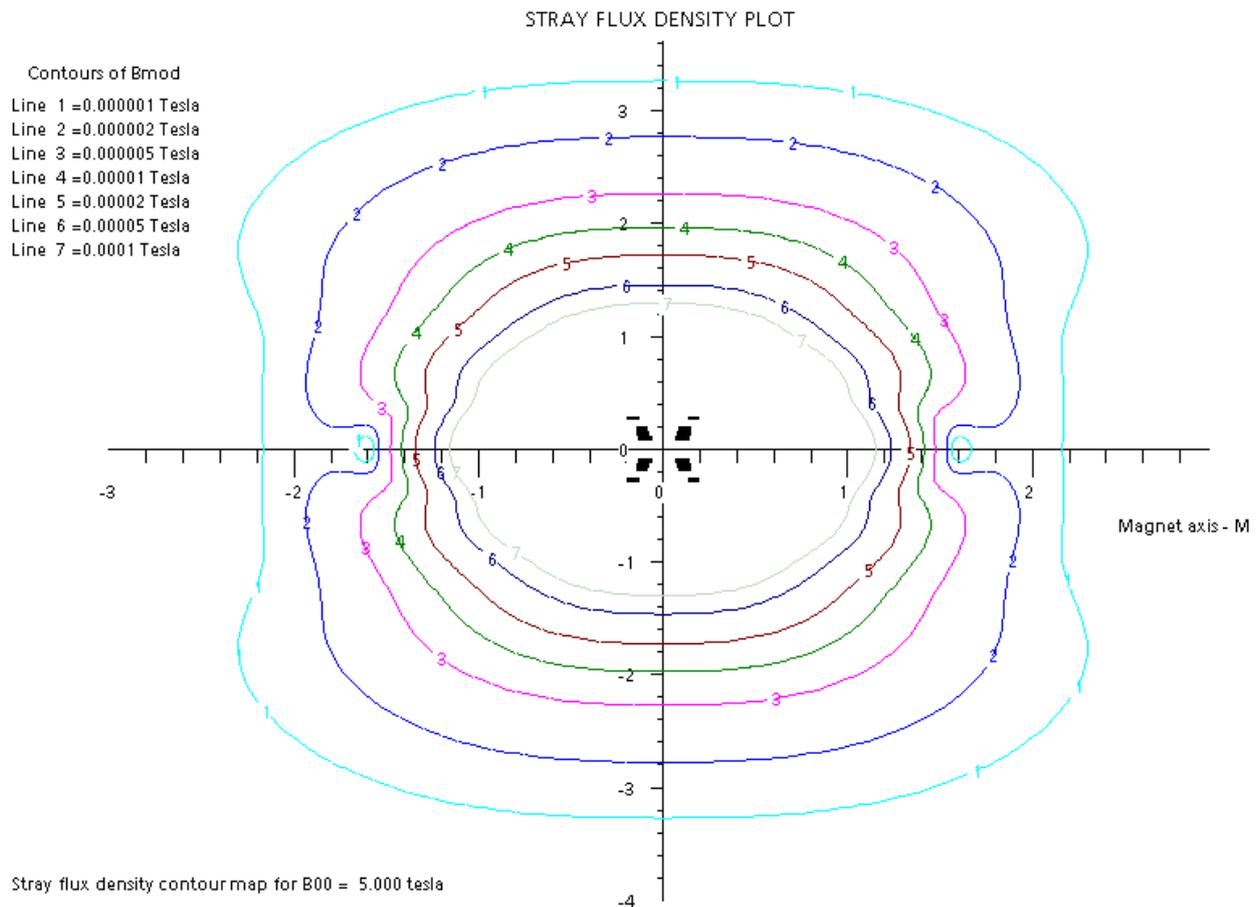


Fig. 2: Line 6 indicates the  $50 \mu\text{T}$  stray flux density. The unit of the axes is 1 metre (image with courtesy provided by Oxford Instruments).

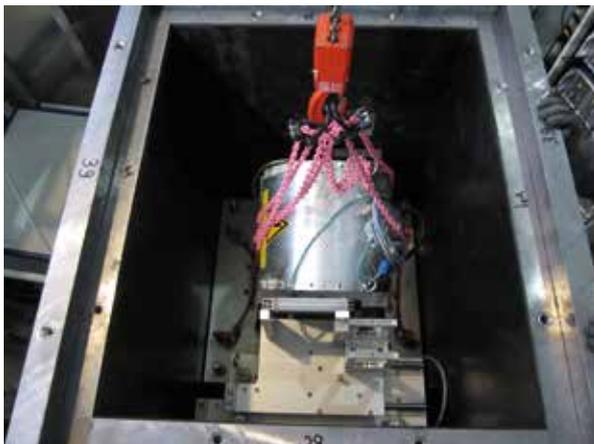
## A New Velocity Selector Housing for RESEDA

The instrument RESEDA uses the **Neutron (Resonant) Spin Echo (N(R)SE)** technique in quasielastic scattering experiments. Rather than employing crystal monochromators, as in three-axes spectroscopy, N(R)SE relies on spin precession of polarized neutrons in magnetic fields. It is sensitive to small energy transfers while using an only coarsely monochromatic neutron beam. High resolution can therefore be achieved without sacrificing a major part of the intensity.



Fig. 1a: RESEDA's velocity selector (image with courtesy provided by Astrium Germany).

Fig. 1b: Installation of the selector in the housing.



Wavelength bands with a monochromaticity  $\Delta\lambda/\lambda_0$  of about 15% (FWHM) can be prepared by means of a neutron velocity selector. The working principle of such a device is similar to that of an aircraft turbine. This can be seen from its rotor-like shape (fig. 1a). Depending on its rotation speed, different mean wavelengths  $\lambda_0$  are obtained because neutrons of too low or high velocity is absorbed in the Boron containing helical grooves. After convolution with the wavelength spectrum fed by the neutron guide, the obtained transmission function  $T(\lambda_0, \Delta\lambda)$  is approximately triangular at the instrument and strongly depends on the orientation of the neutron selector with respect to the beam axis. RESEDA (fig. 2) is located at the neutron guide NL5-S, which also feeds the reflectometer TREFF. The 170 mm high guide is split in two parts: the upper one leads to RESEDA and the lower one to TREFF.

Up to early 2012, the RESEDA velocity selector and the TREFF monochromator were fixed one above the other and housed in the same lead shielding. Due to geometrical constraints, the flange of the selector was partly covering the beam, and so a part of the incoming intensity was lost. In addition, the common housing did not provide the required space for tilting the velocity selector with respect to the neutron beam direction. This restricted the flexibility of the adjustment with respect to the mean neutron wavelength and the width of the wavelength band.

By redesigning the end of NL5-S, including the new selector housing for RESEDA, the situation was substantially improved. The last shutdown period was used to renew the elements which are employed for neutron wavelength preparation. They are now centered in an independent lead shielding (fig. 1b), fixed in a neutron guide gap, about 5 m upstream of the beginning of the N(R) SE instrument region.

The larger allocated space makes it now possible to tilt the velocity selector and to smoothly vary the wavelength used for experiments from 3-10 Å. The option to use short wavelengths is important in order to reach high scattering vector values  $Q$  and, e.g., to access Bragg peaks of many crystals. Finally, sufficient space for a double monochromator option has already been foreseen. In future, a wide range of wavelength widths will be available at RESEDA, opening many doors to elastic and inelastic studies on scattering systems that demand a better  $Q$ -resolution.

*Wolfgang Häußler, FRM II*



Fig. 2: View from the exit of the neutron guide NL5-S towards the instrument. The new velocity selector housing appears in the grey box in the middle.

# Instrumentation

## CASCADE: a high resolution time-of-flight detector

The CASCADE detector was developed by the Heidelberg instrumentation group, which aims for the development of novel systems for neutron detection, especially in the field of **Neutron Resonance Spin Echo (NRSE)** techniques. The counting tube technology was put aside, replaced by solid state neutron converters, of which pure Boron-10 exposed best features.

To overcome the firsthand limitations of an all-solid converter material it was mandatory to set up a system combining different new technologies. The benefits reveal in a high rate capability up to the MHz range, negligible low gamma sensitivity, a x/y spatial resolution of 2.5 mm and z-resolution (time-of-flight) far below 100  $\mu\text{m}$ . For a stack of eight layers an efficiency of about 60% can be reached at 5  $\text{\AA}$ .

### A closer view inside

Boron converts neutrons by inducing an alpha decay releasing a charged particle, having a range of only a few micrometers in the solid. Therefore the medium explicitly has to be thin, decreasing the detection probability of such a single layer to considerably low values.

CASCADE now is the principle of stacking several of these layers on top of one another. This requires a substrate for the converter being transparent for charge and this is fulfilled by the **Gas Electron Multiplier (GEM)** technology - thin foils of a perforated insulator coated on both sides with copper. A precisely adaptable voltage applied between both sides will lead to charge multiplication for those electrons entering the holes (operation in any standard counting gas/ mixture). The charge then drifts towards the readout with its pixel size of 1.6 mm. Furthermore, to locate the exact layer the neutron converted in, the mirror charge induced by the electron cloud traversing the stack is measured. Due to the large energy deposit of the alpha decay, the neutron signal can be discriminated against any background.



Fig. 1: A bare CASCADE system in 20x20 cm<sup>2</sup> with active detection volume (aluminum cover) and readout electronics (below)

### Applications

Following the demands of advanced neutron scattering techniques, CASCADE is able to detect high frequent intensity modulations, a prerequisite for going beyond standard sample interaction times. Spectrometers like RESEDA and MIRA at the FRM II are already equipped with a CASCADE installation.

*Markus Köhli, Universität Heidelberg*

### CASCADE at MIRA and RESEDA

Both instruments offer the **Neutron Spin Echo (NSE)** variant MIEZE (**Modulation of Intensity by Zero Effort**). In order to make it possible to perform low background SANS measurements in strong magnetic fields and of depolarising samples, the beam has to be manipulated before it reaches the sample. MIEZE does this and either a scintillation detector or a CASCADE detector is used. The detector dimensions are 20 cm x 20 cm, divided into 128 x 128 px. The resolution is 2.5-3 mm and the time resolution better than 0.1  $\mu\text{s}$ . See two examples in figs. 3 and 4.

*Wolfgang Häußler, FRM II  
Robert Georgii, FRM II*

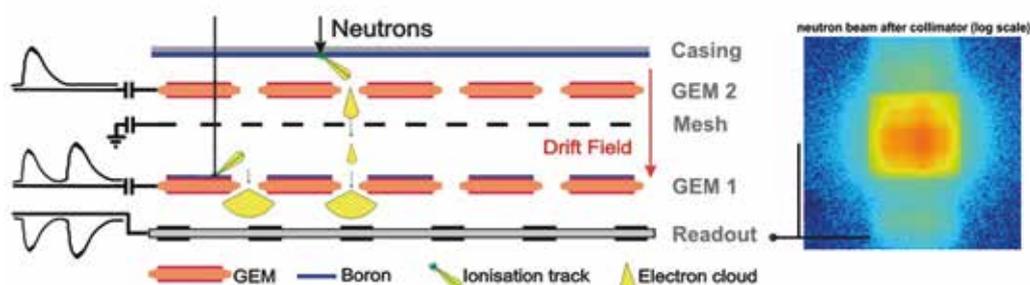


Fig. 2: Schematic cross section of a CASCADE detector and 2D image of a neutron beam (log scale). The schematic shows the minimum unit cell of a detector demonstrating the functional principle.

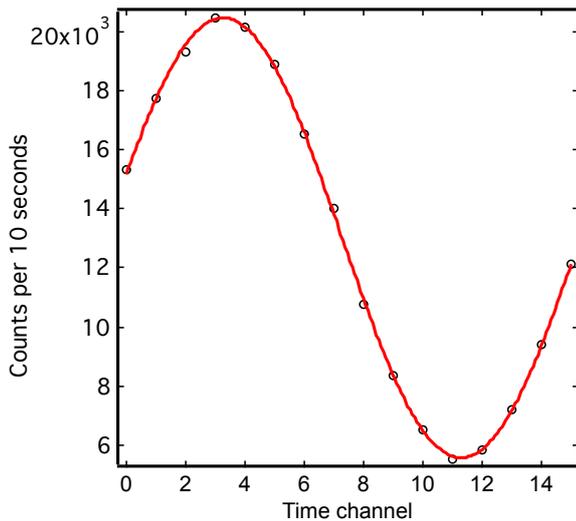


Fig. 3: Oscillating signal measured with a CASCADE detector at RESEDA (one time channel  $\triangleq 0.25 \mu\text{s}$ ).

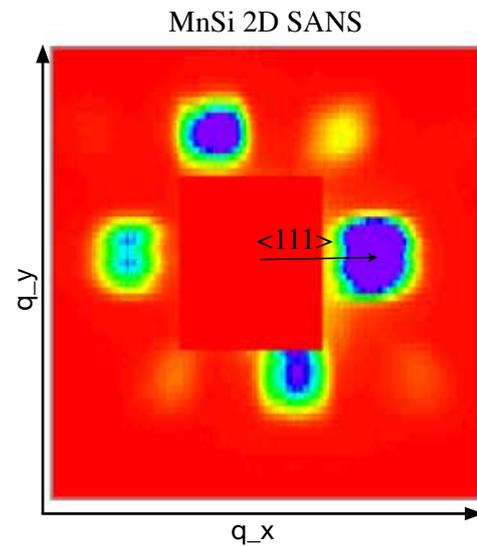


Fig. 4: Small angle neutron scattering signal in zero field from MnSi, taken with a CASCADE detector - the  $64 \times 64$  pixel are well visible. Quasielastic dynamics were measured at the magnetic satellite in  $\langle 111 \rangle$  direction.

## POLI Grows Up

### Biological shielding installed

Since 2010, a BMBF-project (FKZ 05K10PA2) aims for the separation of the diffractometers HEiDi (single crystal) and POLI (polarized) in order to increase the possibilities to use hot neutrons. (only three sources worldwide produce hot neutrons). Each instrument shall get its own beam tube and its own shielded monochromator in the front of hot source SR 9. Thus, two experimental areas shall be created. During the long maintenance break, the beam tube SR9a was prepared for the use at POLI.

Now a further milestone was reached: The new monochromator shielding was installed. Two demands had to be satisfied:

- due to the lack of space, the shielding had to be smaller than that of HEiDi;
- due to the guidelines regarding floor weight limit, it had to be much lighter;
- despite of this, neutrons and gammas had to be blocked more efficiently because of the large beam crosssection on POLI.

The solution was found at ANTARES. The concept of reusable shielding material based on iron powder, boron and paraffin oil cast into steel containers was developed further in order to match POLI's requirements and used as the third layer (fig. 1).

POLI will offer

- thermal neutrons  $2\Theta_M = 41^\circ$  with wavelengths  $0.89 \text{ \AA}$  and  $1.15 \text{ \AA}$ ;
- hot neutrons  $2\Theta_M = 25^\circ$  with wavelengths  $0.55 \text{ \AA}$  and  $0.7 \text{ \AA}$ .

Furthermore an irradiation chamber is integrated within the new shielding. It could be used for example for medical research like developments

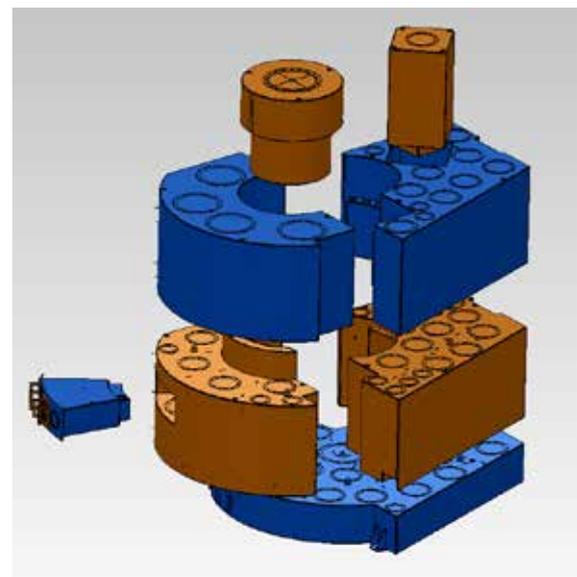


Fig. 1: The reusable shielding material based on iron powder, boron and paraffin oil, recently patented by FRM II, together with boron doped polyethylene and lead layers creates a compact and efficient shielding.



Fig. 2: The new biological shielding at SR 9a (blue-orange) between instruments HEiDi (green-orange) and RESI (rose-red).

in the scope of **Boron Capture Neutron Therapy (BNCT)**.

*Vladimir Hutanu, RWTH Aachen*



# Instrumentation

## New Instrument Open to Users

### MBE system provides thin film fabrication for investigation

**Molecular Beam Epitaxy (MBE)** is a fascinating method to deposit high quality epitaxial thin films. The Jülich Centre for Neutron Science (JCNS) now opens its state-of-the-art MBE system to friendly users who are interested in preparing tailored samples for the investigation with the neutron reflectometer MARIA as well as other neutron scattering instruments or methods.

Epitaxy means the deposition of a crystalline layer on a crystalline substrate, in which the crystal structure of the substrate is reproduced into the film (fig. 1). By this, artificial materials may be fabricated which reveal new properties, like e.g. ferromagnetism at the interface between two anti-ferromagnetic layers or conductivity between two insulating layers.

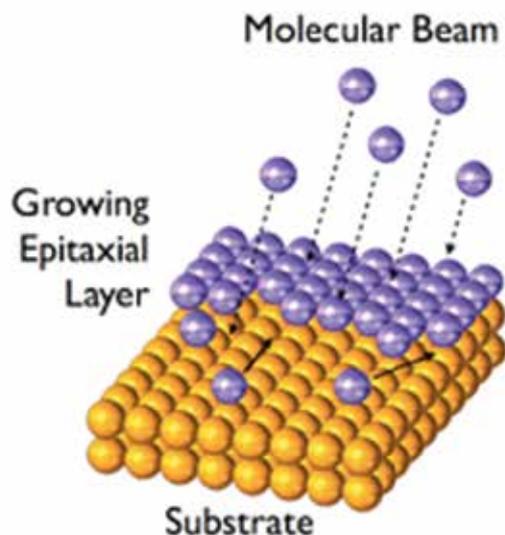


Fig. 1: Molecular Beam Epitaxy (Sketch): Atoms/molecules from a molecular beam adsorb at the surface of the substrate and continue its crystalline structure.

However, high quality thin film growth requires precise control in a multiple parameter space like substrate temperature, evaporation rate (number of atoms arriving at the sample surface per time interval) and the matching of the bulk crystal structures of the involved materials. The residual gas pressure needs special attention. High purity films can only be achieved in **Ultra High Vacuum (UHV)**. As a consequence, sample growth is performed in a UHV chamber with a base pressure of typically  $p \approx 10^{-10}$  mbar (fig. 2). A molecular beam is established inside the chamber either by thermal heating of the material in a crucible (effusion cell) or by heating the material by electron bombardment (e-gun), shown in fig. 3. In the MBE system under operation, there are two e-guns each with

four crucibles and six effusion cells. That means, in principle eight different materials can be evaporated at the same time. Additionally, atomic oxygen is provided by a plasma source. Though the MBE system is designed for the fabrication of transition metal oxides the variety of possible thin films is much broader. Just contact the instrument scientist to check if your idea can be realized!

Via a load lock chamber the substrate is introduced into the UHV system. After pumping it and degassing the sample, the latter is transferred into the main chamber where the thin films are grown. A quality check of the sample surface and in-situ growth control is achieved by **Reflection High Energy Electron Diffraction (RHEED)**, see fig. 4. Additionally, a quartz crystal microbalance is utilized for control of the growth rate. In the buffer line samples can be stored and surface structure analysis with **Low Energy Electron Diffraction (LEED)** (fig. 4) as well as chemical surface analysis with **Auger Electron Spectroscopy (AES)** may be performed.

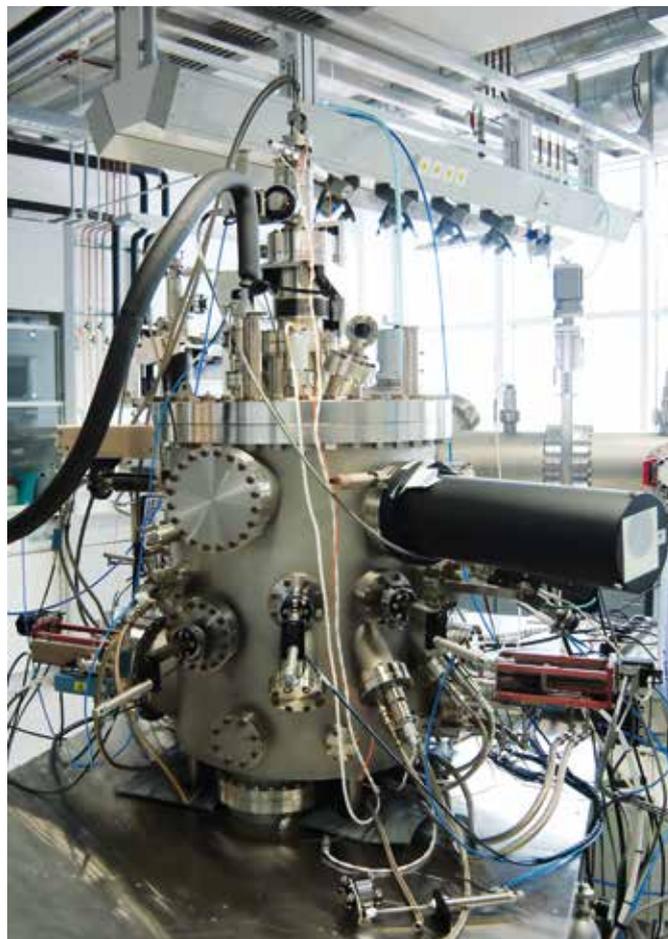


Fig. 2: The oxide Molecular Beam Epitaxy system at JCNS at Garching.

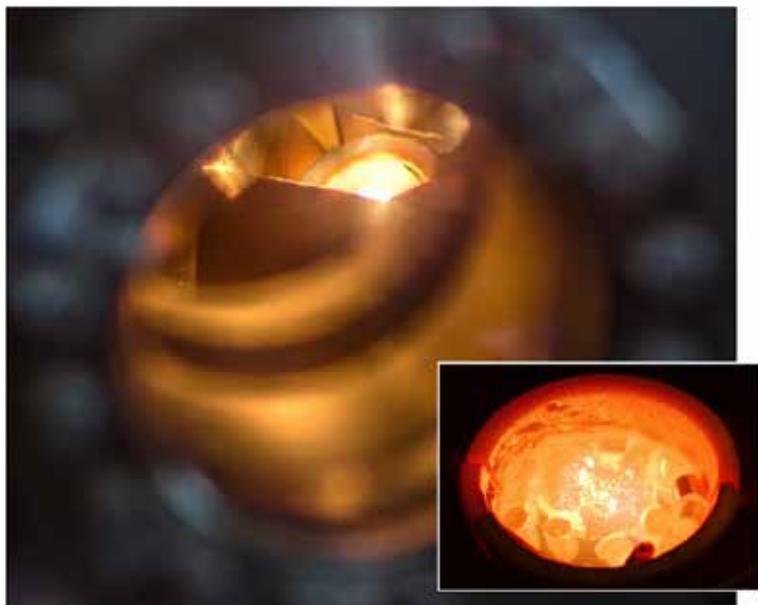


Fig. 3: Inside the MBE: Effusion cell in operation (large picture) and melting gold in the e-gun (small picture).

Examples for high quality samples prepared with the MBE system are given in fig. 4. Additional X-ray reflection measurements of this  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  film on a  $\text{SrTiO}_3(001)$  substrate reveal low surface roughness of 5 Å which is about one unit cell of this compound. The well ordered surface is also visible in the LEED pattern. In agreement with literature the sample is ferromagnetic below 335 K.

For the quasi in-situ neutron reflectivity investigation of MBE grown samples, a special transfer chamber will be developed to transport the thin film sample to the reflectometer MARIA and enable measurements under UHV conditions.

For sample growth, the MBE system may be operated with so-called recipes. Here you programme e.g. shutter opening intervals, evaporation rates and substrate temperature. Once a fabrication procedure for a high quality sample is approved the sample can be easily reproduced with the same quality by applying the recipe.

The JCMS thin film lab also offers an Agilent atomic force microscope and will be upgraded with a setup for measuring the **Magneto-Optical Kerr Effect (MOKE)** as a function of magnetic field and temperature.

We want to address especially those investigators who do not have a thin film fabrication system at hand. Two operation modes are offered. For remote access, the instrument scientist will grow the sample for you. Of course, this is only possible in cases where a recipe is at hand. Alternatively, in a collaborative approach you join us in Garching for sample growth. This mode will be necessary, if research on the proper growth conditions is required.

From now on we offer:

- Technical guidance about the feasibility of your ideas
- User initiated MBE growth of thin films
- Sample characterization by AFM and MOKE

Hope to welcome you soon!

*Sabine Pütter, JCMS*

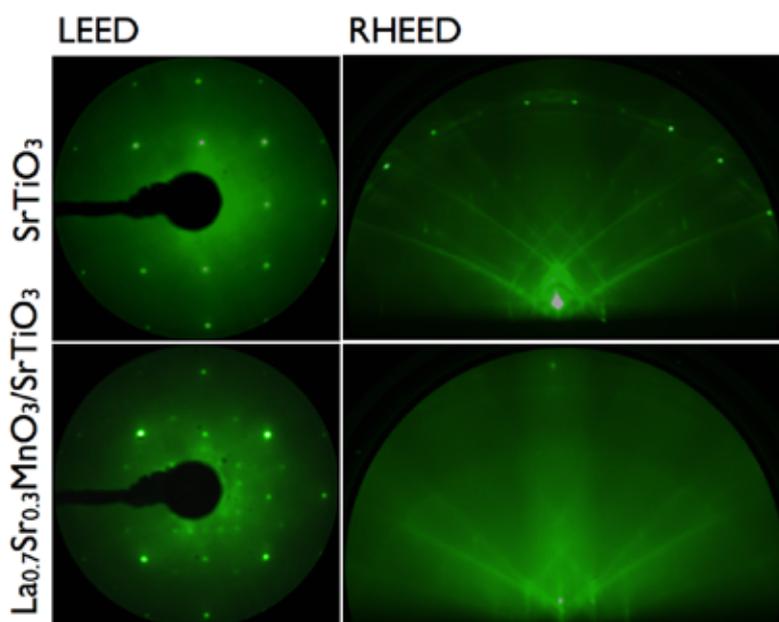


Fig. 4: Sample characterization with electrons in k-space: (left) Low Energy Electron Diffraction patterns at 100 eV and (right) Reflection High Energy Electron Diffraction patterns at 15 keV of (top)  $\text{SrTiO}_3$  and after deposition of 18 nm  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  on top (bottom).

## Transmission Electron Microscopy Facility at the JCNS



Fig. 1: JEM-FS2200 Transmission electron microscope at JCNS@FRM II.

With the neutron instrumentation at the FRM II, in particular small angle neutron scattering, reflectometry and macromolecular crystallography, structures in the range from 1 nm up to several hundred nm are investigated. In soft matter and biology the contrast between hydrogen and deuterium is used to gain deep and quantitative insights about the shape and interactions of the objects forming the investigated structure, e.g. various phases of microemulsions, polymer aggregates in solution, micelles, colloids, fillers in polymers, block copolymer phases, protein molecules and aggregates and many other systems.

The neutron methods enable observation under functional conditions (e.g. in liquid solution) and yield quantitative information on pair distribution functions that are directly related to thermodynamical descriptions.

However, the “phase problem” inherent to all scattering experiments remains and makes difficult the investigation of complicated, asymmetric shapes. Knowledge on these shapes, their type and symmetry would often be vital to find the proper model to describe neutron scattering results and extract further quantitative information from them.

To overcome this limitation, imaging techniques, i.e. microscopy, is needed. The optics of a microscope preserves phase information and yields images in ‘real space’. The closest option covering the spatial range of interest is electron microscopy. **Transmission Electron Microscopy (TEM)** may yield real space pictures of soft matter aggregate, microemulsions and all the other systems listed above, virtually it may complete and enhance any SANS investigation on soft matter investigation.

On the one hand, with TEM, we are able to extract information in the real space about size measurements and distribution of particles, self assembly systems and aggregates as well as shape.

On the other hand SANS is a non-destructive method providing structural information in the reciprocal space averaged over all particles of different sizes with high statistical accuracy. Additionally quantitative information about roughness and intermolecular distances can be extracted and indirect information concerning average radius and periodical distances by performing a Fourier transform in transmission electron microscope data.

Moreover, in TEM, diffraction patterns can be obtained.

Neutrons have the advantage of being sensitive to light elements, in particular hydrogen and deuterium which remains invisible in TEM and X-ray small angle scattering, especially for porous materials which scatters strongly. Scattering profiles, in momentum transfer ( $q$ ) range covering four orders of magnitude and with scattering intensity of more than ten orders of magnitude, can be used to determine the internal structure and the microporosity.

Both techniques allow to detect structural changes occurring in the relatively large scale structures on the micro- and nano-scale. One main advantage in SANS is the contrast variation technique which allows, by using specific ratio of  $D_2O/H_2O$  for solution samples, to study multi-component systems.

With TEM, the energy filter and the energy loss method allows to get information about atomic composition of the observed system.

The energy filter is an additional way to enhance contrast in complementary to thickness dependent amplitude contrast, the small angle phase contrast and the diffraction contrast for crystal structure investigation.

The JCNS now offers the combination of neutrons and TEM to its users. Therefore a TEM laboratory has been installed around a **Field Emission Gun (FEG)** 200 kV instrument with Omega energy filter. Unlike for neutron experiments the soft matter

samples have to be investigated either in dried or frozen state (Cryo-TEM) to be able to work in the necessary vacuum and to suppress blurring motion of the object as well as radiation damage. Also the samples have to be very thin (max~100 nm). Because such investigations require sophisticated sample preparation different from the needs for e.g. a SANS experiment, the TEM laboratory comprises an extended suite of preparation equipment. Prior to investigation, grids are prepared by carbon coating and glow discharge in order to make them hydrophilic via a sputtering device. Aggregates and particles in solution can be di-



Fig. 2: Automatic plunge freezer.

oblique evaporation of Pt/C or Ta/W are prepared. The TEM investigation is performed on these replicas, a suitable technique to capture structures in a microemulsion. Finally for bulk (polymer) samples, frozen blocks etc. a cryo-ultramicrotome is available.

Users will be supported by JCNS scientists (M.S. Appavou) to conduct the suitable preparation and TEM investigation.

*Marie-Sousai Appavou, JCNS*



Fig. 3: Docking station and Carbon sputtering coater.

rectly investigated by shock freezing of thin liquid films (cryo-plunge) that are then transferred into the microscope.

Also freeze fracture technique is available and supported; here replicas of fracture surfaces by

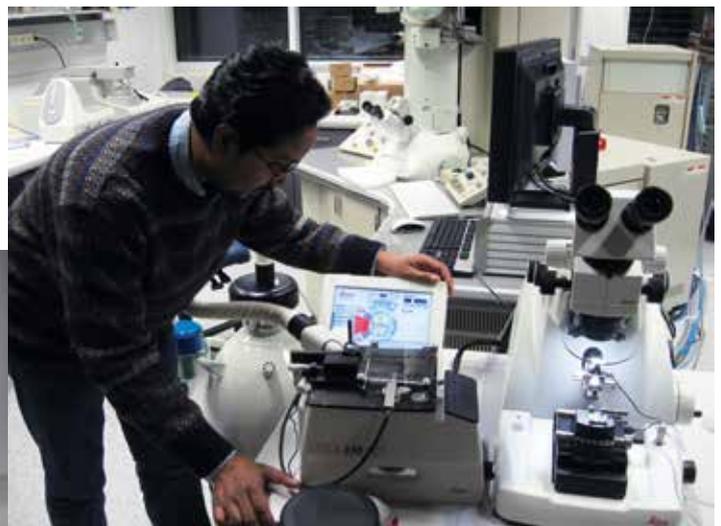


Fig. 4: High quality ultramicrotome for precise room temperature and cryo sectioning. M.S. Appavou is planning to install the specimen cryo-sectioning chamber instead of the room temperature specimen sectioning chamber.

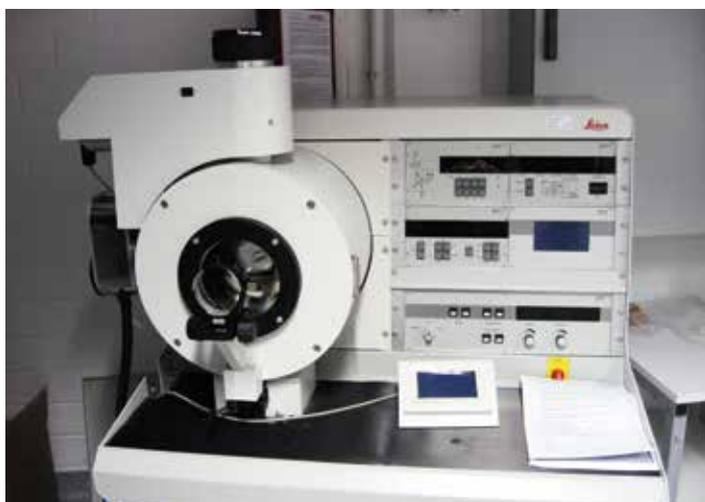


Fig. 5: Freeze fracture and etching system.

# Instrumentation

## From Chocolate to Steel

### The Hard-Soft-Matter Lab



In collaboration with the Helmholtz Zentrum Geesthacht the Technische Universität München (TUM) runs a Hard-Soft-Matter Lab at the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II).

The lab is currently equipped with several instruments and facilities for sample preparation techniques which allows preparing samples for neutron scattering measurements as well as complementary analytic methods to analyse them.

From the analytic part of the lab we would like to point out the **Small Angle X-ray Scattering (SAXS)**, the **Differential Scanning Calorimetry (DSC)** and **Laser Diffraction Particle Analyzer (LD-PA)** instruments.

The SAXS instrument (Hecus S3 MicroPix) is featured with a Pilatus 2D detector (84 x 34 mm<sup>2</sup>, q-range 0.1 - 60 Å<sup>-1</sup>) and an additional 1D wide angle detector (2 $\theta$  18° - 30°). It allows SAXS, GISAXS and WAXS measurements. Liquid as well as solid samples (in a limited way) can be cooled down to -20°C and annealed up to +300°C in situ e.g. for temperature scans.

Two scanning calorimeters for measuring phase transition temperatures and heat capacity respectively are available: A Perkin Elmer DSC 8500 system covering a temperature range of -180°C up to 650°C with an accuracy of +/-0.05°C and a high resolution  $\mu$ DSC Evo 3 system (-20°C...



Fig. 2: Loading a sample into the furnace.

120°C) providing an accuracy of +/-0.001°C.

The laser diffraction particle analyzer (Beckman Coulter LS13320) allows particle size measurements in the range of 20 nm – 2 mm from suspensions using Fraunhofer diffraction and **Polarization Intensity Differential Scattering Analysis (PIDS)**.

A high resolution digital optical microscope with considerably filter and contrast techniques

completes the analytical equipment of the lab.

The part of the lab reserved for sample preparation offers a water cooled corundum saw (ATM Brillant 200) to cut hard samples and a polishing machine (ATM Saphir 520). Techniques for cold embedding and a machine for hot embedding (ATM Opal 410) are on hand as well. Thereby samples (e.g. powder, solid pieces etc.) can be fixed into various resins (conductive, shrink-free, transparent...) in respect to subsequent polishing for SEM, EDX and microscopy analytics.



Fig. 1: Loading a sample into the SAXS instrument.

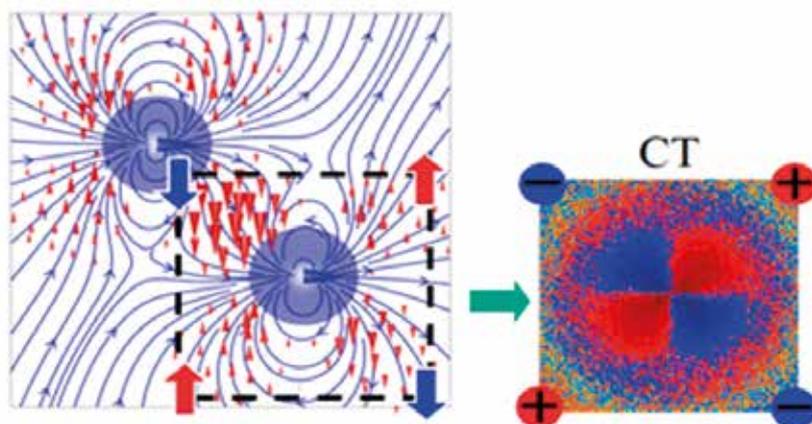
A chemical hood for a little chemistry, a vacuum drying oven (up to 200°C) and a high temperature corundum tube furnace (up to 1500°C, Gero) completes the preparation possibilities.

Current projects for example are SAXS analysis of LiFePO<sub>4</sub> battery materials and nanoparticle suspensions as Ni<sub>3</sub>Si core shell, Au- and SiO<sub>2</sub>-nanoparticles, TiO<sub>x</sub> nanoparticle embedded in medical implant polymers, eye-drop solutions, DNA-Triglyceride drug carrier and growth mechanism of ZnO particle suspensions for Grätzel solar cells. The DSC is used for investigation of alloys like Al-FeNi, NiMnInCo-memory alloy and UMo as well as for di-block copolymers.

Armin Kriele, HZG-GEMS

## Micromagnetic Theory & SANS

Garching, June 21<sup>st</sup>-22<sup>nd</sup>



Results of a micromagnetic simulation.

Left image: Real-space magnetization distribution around two nanoparticles (violet spheres) that are embedded into a magnetic matrix of different magnetization. Shown is the perpendicular component of the magnetization (red arrows). The applied-field direction is horizontal ( $B_0 = 0.3$  T). Blue lines: magnetodipolar field distribution.

Right image: Computed 2D Fourier image of the cross term  $CT \propto M_x(\mathbf{q}) M_y(\mathbf{q})$  revealing an alternating sign of this term at the quadrants of the detector.

From June 21<sup>st</sup> - 22<sup>nd</sup>, 2012, the workshop *Micromagnetic Theory & SANS* was held at the FRM II at Garching.

Experimentalists and theorists met in order to discuss the state-of-the-art in the field. Micromagnetism is a mesoscopic phenomenological theory designed to compute the equilibrium magnetization state of an arbitrarily shaped ferromagnetic body, when the applied magnetic field, the geometry of the ferromagnet and all materials parameters are known.

Recent progress in the development of a micromagnetic simulation methodology permits the computation of the magnetic microstructure and of the associated magnetic SANS cross section of a wide class of magnetic materials. The decisive advantage of this approach resides in the possibility to study the individual magnetization Fourier contributions to the total magnetic SANS cross section (shown in the figure above), rather than their sum, which is generally obtained from experiment.

The workshop programme consisted of invited and contributed talks, which were divided into three sessions (*Numerical Simulations, Instrumentation, Spin Structures*). After a brief welcome by Winfried Petry, the scientific director of FRM II, Andreas Michels (University of Luxembourg) gave an overview talk on magnetic SANS on nanomagnets. An introduction into the basics of micromagnetism and its applications to magnetic SANS experiments was given by Dmitry Berkov and Sergey Erokhin (both INNOVENT, Jena), followed by a talk of Frédéric Ott (CEA/CNRS) on the numerical calculation of magnetic form factors of complex shaped nanoparticles. With "his" new 17 T magnet, Ted Forgan (University of Birmingham) described possibilities to explore new areas for the interaction of high magnetic fields with superconductors, magnetic materials and colloids in liquid suspension. Anna Sokolova (ANSTO) introduced

the new time-of-flight SANS instrument BILBY at ANSTO, while Kathryn Krycka (NIST) reported on recent developments regarding SANS polarization analysis at NIST.

In the afternoon of the first day the workshop attendees made an excursion to the neutron guide hall of FRM II; during this visit particular attention was paid to the new SANS-1 instrument.

The second day of the workshop consisted of a very interesting session devoted to spin structures, which was mostly run by scientists of the Technische Universität München (TUM): Sebastian Mühlbauer gave a seminar on ferromagnetic correlations of the almost antiferromagnetic Dzyaloshinsky-Moriya helimagnet  $\text{Ba}_2\text{CuGe}_2\text{O}_7$ , Christian Pfeleiderer delivered an overview talk on the emergent electrodynamics of skyrmions in chiral magnets, and Jonas Kindervater explained how spherical neutron polarimetry can be employed in order to study the transition from heli- to paramagnetism in MnSi. The workshop was rounded off by a talk of Artem Feoktystov (JCMS) on the SANS analysis of magnetic fluids using contrast variation.

About 30 researchers from seven countries attended the one and a half day workshop.

In conclusion, taking into account the qualitative methodical progress achieved by numerical simulations of magnetic neutron scattering cross sections, the participants agreed to establish a close cooperation between experimental and theoretical groups. This cooperation should lead to a decisive improvement of our understanding of magnetic SANS experiments, especially on materials with highly complicated magnetic structures such as nanocomposites or helical magnets.

*The organizers:*

Andreas Michels, University of Luxembourg  
Dmitry Berkov and Sergey Erokhin, INNOVENT Technologies, Jena  
André Heinemann, HZG-GEMS



## 16<sup>th</sup> JCNS LabCourse

Jülich and Garching, September 3<sup>rd</sup>-14<sup>th</sup>

The 16<sup>th</sup> JCNS Laboratory Course Neutron Scattering took place September 3<sup>rd</sup>-14<sup>th</sup>, 2012. Like in the previous years, this annual lab course was held at two locations: at Forschungszentrum Jülich for the lecture part and at the neutron source FRM II in Garching, close to Munich, for the experiments.



Students asking curious questions about the analysers of the neutron backscattering spectrometer SPHERES.

The lab course is open to students world-wide of physics, chemistry, and other natural science. Participation is free of charge for the selected students, and travel expenses are reimbursed for foreign students. The course is part of the curricula of the Universities of Aachen and Münster. Funding came from Forschungszentrum Jülich with support of the Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy (NMI3), the European Soft Matter Infrastructure project (ESMI), and the SoftComp Consortium.

The first week of the neutron scattering course is always dedicated to lectures and exercises encompassing an introduction to neutron sources and presenting scattering theory and instrumentation. Furthermore, selected topics of condensed matter research were addressed.

In the second week, eleven instruments at FRM II were made available for students' training, including the neutron spin-echo spectrometer J-NSE, the backscattering spectrometer SPHERES and the small-angle scattering instruments KWS-1 and KWS-2. Those world-class instruments were provided by JCNS, RWTH Aachen, University Darmstadt, University Göttingen, LMU Munich, and TU München.

This year 59 students were selected from 143 applicants. For the first time in

the history of the course the majority of the students were from chemistry (28), followed by physics (24) and material sciences (7). 28 foreign students came from a total of 14 countries. This year we were glad to welcome the first students from Palestine, Brazil, and Taiwan. The participation of female students was 37%.

During the first week lectures were held in combination with tutored exercises and the syllabus was condensed. This new concept was introduced last year and proved successful again.

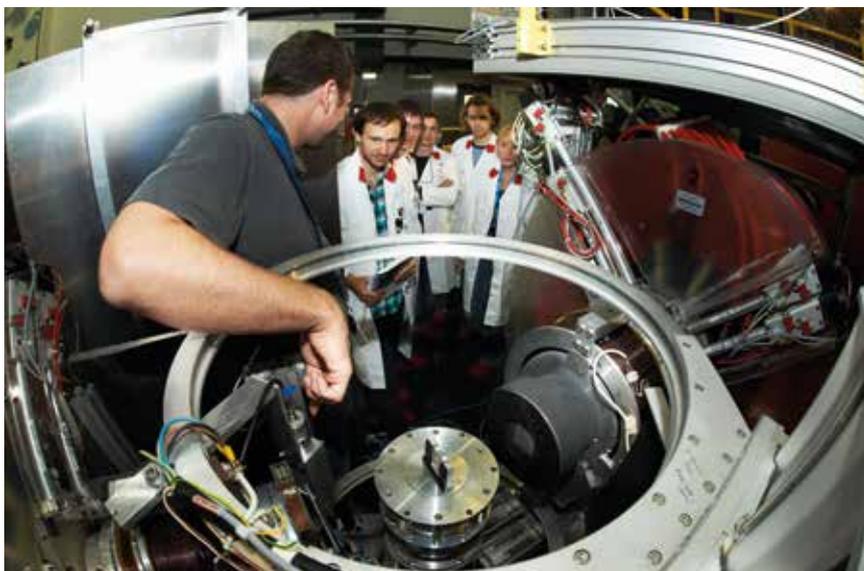
In the second week the performance of the neutron source was flawless, so that experiments could be carried out all five days. Except for the usual one, that passports turn out to be the most volatile objects in the world when it comes to access control, no major problems hampered the practical part of the course.

The programme was completed by a welcome party in Jülich and a farewell party in Garching. For the latter, a students' café was opened in the evening allowing the students (and some of the tutors) to enjoy food, drinks and dance. In order to offer the students physical training in another sense, soccer, volleyball, and slacklining were the topics of choice on one of the afternoons of the second week.

The next JCNS laboratory course will take place September 2<sup>nd</sup>-13<sup>th</sup>, 2013, following the same concept having the first (lecture) week in Jülich and the second (experimental) in Munich. You are cordially invited to submit applications. In spring 2013, more details will be posted at

[www.neutronlab.de](http://www.neutronlab.de)

Reiner Zorn, JCNS/ Forschungszentrum Jülich



Olaf Holderer explaining the subtleties of the field distribution at the sample position of the neutron spin-echo spectrometer J-NSE.

## German Conference on Neutron Scattering

Bonn, September 24<sup>th</sup>-26<sup>th</sup>



Research with neutrons was the common ground of 170 scientists from different disciplines who gathered at the *German Neutron Scattering Conference* in Bonn September 24<sup>th</sup>-26<sup>th</sup>, 2012. Scientists from particle and condensed matter physics, chemistry, biology, materials sciences, engineering sciences, right up to geology and cultural heritage used the opportunity to present their results, to exchange ideas and to gather methodological inspiration. The meeting was organized by the Jülich Centre for Neutron Science (JCNS) of Forschungszentrum Jülich on behalf of German Neutron Research Committee (KFN).

The session on the scientific perspectives for the ESS was a highlight of the conference and clearly demonstrated how powerful neutron research is and how much it will benefit from the unprecedented advances offered by the ESS. The planning of ESS is now gaining momentum not only in Lund, but also among the German neutron scientists.



Conference dinner in the castle Godesburg.

State-of-the-art instruments at world-class large-scale facilities enable work on exciting scientific questions making neutron research attractive to young scientists. To recognize this, the KFN awards the Wolfram-Prandl-Prize to young researchers using neutrons for their studies. This year, Dmytro Inosov from the Max Planck Institute (MPI) for Solid State Research received the prize for his outstanding work on high-temperature superconductors. Inosov's results indicate that the high-temperature superconductivity in iron pnictides, unlike in conventional superconductors, is due to magnetic excitations. This is an important basis for future theoretical and experimental work. The conference dinner in the castle Godesburg added a festive touch to the DN-2012. The accommodation in Gustav-Stresemann-Institut facilitated the exchange of ideas on the sidelines not only among the participants but also with decision-makers in the BMBF (German Federal Ministry of Education and Research). We thank the organizers who made the success of this meeting possible and are looking forward to the upcoming SNI-2014.

Dmytro Inosov, Wolfram-Prandl-Prize laureate, during his presentation.



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*Karin Griewatsch, Universität zu Kiel, KFN*

# Events

## 26<sup>th</sup> TANGO Collaboration Meeting

Herrsching, September 25<sup>th</sup>-27<sup>th</sup>

The 26<sup>th</sup> TANGO collaboration meeting was organized by the FRM II for the first time. The meeting is open to everybody interested in using TANGO (a distributed network based instrument control system) including commercial companies, and it is organized alternating by one of the members of the TANGO group. Its members signed the so called **Memorandum of Understanding (MoU)** which handles the aspects of the collaboration. The European Synchrotron Radiation Facility (ESRF) and some national Synchrotron sources like SOLEIL (France), Elettra (Italy), ALBA (Spain), DESY (Germany), MaxLab (Sweden), and SOLARIS (Poland) as well as the neutron source FRM II (Germany) belong to this group.

The around 30 participants informed each other about the current state of use at their facilities and the plans for the future. The current developments in the TANGO core libraries, and of tools which could be interesting for others were also presented.

After fruitful and lively discussions at the shores of lake Ammersee, twenty participants took the chance to tour the FRM II at Garching and got really impressed by the FRM II's technique and the hosted scientific experiments.

*Jens Krüger, FRM II*



The participants visiting the famous Kloster Andechs before they joined the conference dinner.

## JCNS Workshop on Soft Matter and Biophysics

Tutzing, October 8<sup>th</sup>-11<sup>th</sup>



The Jülich Centre for Neutron Science (JCNS) and the Donostia International Physics Center (DIPC), Spain, jointly organized the 2012 workshop *Trends and Perspectives in Neutron Scattering for Soft Matter and Biophysics* held in Tutzing, Germany, on October 8<sup>th</sup>-11<sup>th</sup>.

75 attendees joined this event for 42 presentations and 16 posters. The combination of the conference's charming location at the Protestant Academy in Tutzing in conjunction with the highly motivated attendees from Europe, USA, and Japan created a stimulating atmosphere which resulted in numerous interesting discussions throughout the workshop.

Topical sessions such as *Advanced Instrumentation for Soft Matter Research*, *Physics of Life*, *Soft Materials for Energy*, *Kinetic Processes*, *Sustainable Polymers*, *Functional Materials*, *Synergies*

*between Neutrons and Simulation* and *Nanostructured Systems* formed the structure of the workshop.

Recently developed nanodevices may play a key role in future strategies for tumour therapy. In this field, neutron scattering is of fundamental importance for an understanding of the key processes. Soft matter materials were described as key enablers for future renewable energy sources and biofuels. It was clearly demonstrated that even for very complex systems, modelling is becoming more and more important to fully exploit neutron scattering experiments and deepen basic understanding.

The rapid progress in the field of soft matter research and biophysics results in an ever increasing challenge to extend the limits of existing neutron instrumentation and to start building specially-adapted high performance instruments at both existing and future neutron sources such as the European Spallation Source (ESS).

JCNS and the Institute of Complex Systems (ICS) contributed a total of eight oral presentations and eleven posters, underlining the key role of JCNS in neutron scattering-based soft matter research and advanced instrumentation.

The next JCNS workshop will focus on magnetism and will be held in Tutzing on October 7<sup>th</sup>-10<sup>th</sup>, 2013.

*Rainer Bruchhaus, JCNS*

## Hot Coffee, Ultra-Cold Neutrons

### Lively participation at a series of public events

In September, the Research Neutron Source Heinz Maier-Leibnitz (FRM II) for the first time participated in the well-established and highly successful event *Café & Kosmos* organized by the Universe Cluster, the European Southern Observatory and three Max Planck Institutes. In a relaxed atmosphere and with no technical support (no projector, no PowerPoint), scientists present their research projects – for the price of a cup of coffee or a glass of wine. On September 11<sup>th</sup>, Peter Fierlinger from the Universe Cluster discussed with participants why ultra-cold neutrons can unravel the mystery of antimatter, and on October 9<sup>th</sup>, Christoph Huguenschmidt talked about cosmic positron sources and the benefit of positrons in materials research. The local pub Vereinsheim in Schwabing was fully packed with about 70 participants and each presentation was followed by a long discussion.

Every three years in mid-September, the trade show *Garching Herbsttage* with local companies and businessmen is held in Garching. Traditionally, the Research Neutron Source is also present at a very prominent stand at the entrance of the community centre. In order to increase the attractiveness of the stand and to better get into contact with the audience, the FRM II presented a new throwing game for children. With balls, “free neutrons”, they could try to hit the target, the “nuclei” of a NaCl crystal lattice. Great success!

At the traditional *Open Day* on the Campus Garching, this year held on October 27<sup>th</sup>, the visitors again showed great interest in a tour of the Research Neutron Source. From 11 a.m. on, a long queue formed at the registration desk, and in the early afternoon all tours were fully booked. 500 visitors took advantage of the opportunity. In addition presentations by scientists in the neighbouring Physics Department, the audience was able to learn more about the scientific issues researchers are dealing with at the Neutron Source.

Finally, from November 12<sup>th</sup> to 15<sup>th</sup>, the FRM II members of the battery project ExZellTUM presented the benefits of neutrons for battery research at the international trade fair for batteries, energy storage and innovative production ees, a special exhibition within the *electronica* trade show at Messe München.

*Petra Riedel, FRM II*



Shortly after the beginning of this year's *Open Day*, a long queue formed at the registration desk for a tour of the Neutron Source.



Lively discussions followed the talks of Christoph Huguenschmidt (l.) and Peter Fierlinger (r.) at the *Café & Kosmos*.



Project coordinator Ralph Gilles during his talk on the benefits of neutrons in battery research at the international trade fair ees presenting the ExZellTUM project.



Success! Assisted by Jürgen Neuhaus, a junior scientist (l.) had much fun with trying to hit “atomic nuclei” with “free neutrons” at the FRM II stand at the *Garching Herbsttage*. Even mayoress Hannelore Gabor (r.) tried to be a neutron scientist, but only won a consolation prize presented by Rainer Bruchhaus.



## The NMI3 Deuteration Lab

### Combining neutrons and NMR spectroscopy at TUM

An international consortium of the European NIM3 research network coordinated by FRM II/ TUM develops efficient and optimized biochemical protocols for the deuteration of proteins. This deuteration lab (DLAB) project will open new ways in using neutron experiments in structural biology.

A major challenge in life sciences is determining the three-dimensional structures of proteins and protein complexes at atomic resolution. Such information is required to provide a detailed understanding of the molecular mechanisms of essential cellular processes underlying health and disease. The availability of these structures is also a prerequisite for the rational design of drugs.

However, conventional methods such as X-ray crystallography often face difficulties in the structure determination of these complexes because of their dynamic nature. **Nuclear Magnetic Resonance** spectroscopy (NMR) can provide information about domain arrangements, binding interfaces and is especially suited to characterize the protein dynamics at time scales ranging from picoseconds to days. However, for larger protein complexes NMR suffers from low sensitivity, and often only sparse structural data can be obtained that may not be sufficient to reveal high resolution structural details.

Here, **Small Angle Neutron (and X-ray) Scattering** (SANS/SAXS) data can provide powerful complementary information. Computational tools are available to assemble multi-protein complexes by combining data from NMR, SAXS and SANS data, thus opening new ways in structural biology. Especially the combination of NMR and neutron scattering data is interesting as the same samples can be used for NMR and neutron experiments,

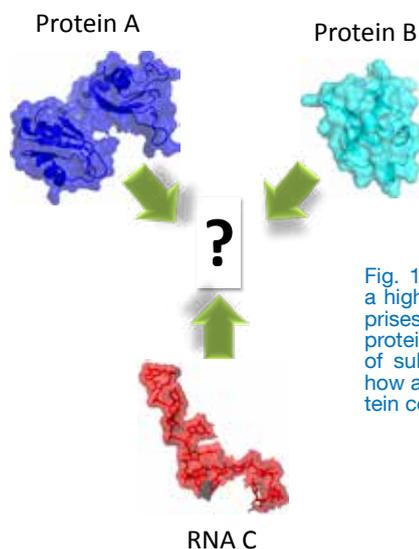


Fig. 1: A protein complex forms a higher order assembly. It comprises of three components, two proteins and one RNA. Structures of subunits may be known, but how are they arranged in the protein complex?

and both techniques require  $^2\text{H}$ -isotope labeling of the protein complexes studied. One of the projects in the NMI3 network develops sophisticated biochemical methods for  $^2\text{H}$ -labeling of proteins for neutron experiments.

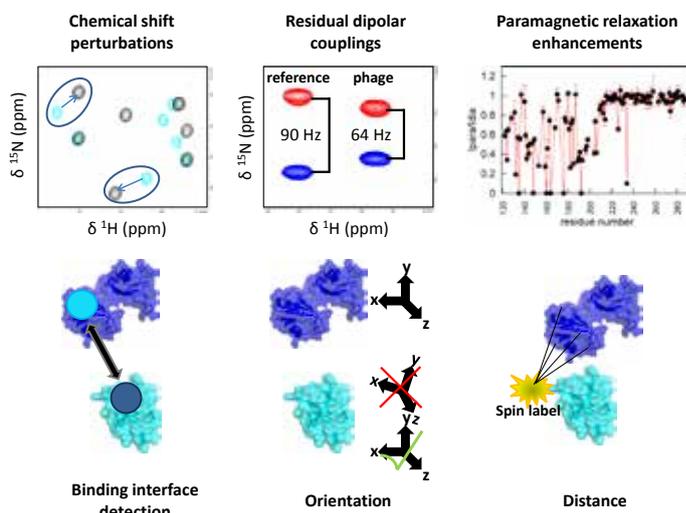


Fig. 2: Nuclear magnetic resonance (NMR) spectroscopy provides structural information about the protein complex.

Left: NMR resonance frequencies of nuclear spins in the binding site are shifted.

Centre: Residual dipolar couplings measured in absence and presence of an alignment medium (phages) provide information about relative domain or subunit orientation.

Right: A paramagnetic probe (yellow) attached to one of the complex subunits attenuates signals of residues in spatial proximity. The ratio of NMR signal intensities in the paramagnetic protein and a non-paramagnetic reference provide long-range (up to 2 nm) distance information.

Nowadays high-resolution crystal or NMR structures are often available for individual domains or subunits of a complex at the Protein Data Bank ([www.ebi.ac.uk/pdbe](http://www.ebi.ac.uk/pdbe)). For example, structures of the A and B subunits/ domains of a complex consisting of two proteins (A and B) with a single-stranded RNA (C, fig. 1) may be available. But how are these components assembled to a larger biologically relevant complex? NMR provides various experimental data to determine the arrangement of individual components. Binding interfaces can be mapped from chemical shift perturbations, i.e. changes in the NMR resonance frequencies of nuclear spins of residues involved in a binding interface shift upon complex formation. Residual dipolar couplings harbour information about the relative orientation of individual domains of a complex, or paramagnetic relaxation enhancements can provide distance measurements between subunits (fig. 2). However, structure calculations based on such sparse data do not always provide unambiguous structural models of the protein complex.

Small angle scattering data can then be used to validate and score the structural models. The use of SANS is especially valuable as it provides information about the orientation of individual subunits within the complex using “contrast matching”, whereas SAXS only gives insight into the overall shape of the complex.

For example, if all hydrogens of protein B are exchanged to deuterium, and the sample is measured in 70% D<sub>2</sub>O buffer, the RNA is rendered invisible and a high scattering contrast is obtained for protein B and protonated protein A (fig. 3). At 42% D<sub>2</sub>O content of the solvent buffer, the scattering contrast is matched between the protonated protein A and the solvent and thus information about the shape of protein B and RNA is obtained. In a multi-domain protein, where several structural domains are connected like pearls on a string, SANS contrast matching can be employed by <sup>2</sup>H-labeling of one of the domains but not the others using segmental domain labeling. The exchange of protons to deuterons in proteins is easily achieved by growing the *E.coli* bacteria that overexpress the recombinant protein in a buffer containing deuterated glucose and 100% D<sub>2</sub>O.

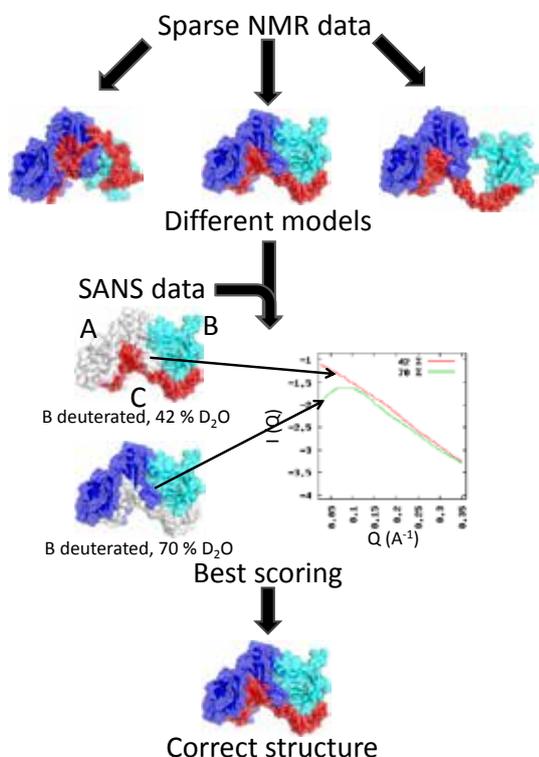


Fig. 3: Different structural models of the protein complex are obtained from structure calculations based on sparse NMR data. The correct structure can be identified based on the best fits to SANS data measured at different contrast matching conditions (42% and 70% D<sub>2</sub>O, components being matched with bulk solvent are coloured white) and deuteration.



Fig. 4: Janosch Hennig navigating an 800 MHz (18.8 T) NMR spectrometer (bottom). The combination of NMR and SANS data (which can be recorded at FRM II) boosts structural biology research to new heights.

In recent years, the unique information provided by neutron experiments, especially from SANS, is increasingly appreciated by the structural biology community and integrated approaches combining crystallography, NMR, electron microscopy and small angle scattering data enable studies of challenging protein complexes. Munich harbours outstanding and unique infrastructures for these studies. For example, FRM II for SANS measurements and the Bavarian NMR Centre (BNMRZ, [www.bnmrz.org](http://www.bnmrz.org)) with numerous high magnetic field NMR spectrometers (ranging from 11.7 to 21.1 Tesla magnetic field strengths) are located at the TUM Campus Garching in immediate vicinity (fig. 4). In the future, further developments in sample preparation, NMR methods and neutron experiments will greatly expand the scope of structural biology in basic and biomedical research.

*Michael Sattler, TUM  
Hennig Janosch, Helmholtz Zentrum München*



# Science & Projects

## Raising the Pressure

### Installing SAPHiR in the new neutron guide hall east



Fig. 1-2: SAPHiR starting its way to Garching in Mainleus, Oberfranken. Above: The positioning table. Below: Loading of the 50 ton multianvil press.



Fig. 3: Last preparations in the neutron guide hall east at Garching.

The **Six Anvil Press for High pressure Radiography and Diffraction instrument SAPHiR** is dedicated to time-of-flight neutron diffraction studies and high resolution neutron radiography at extreme pressure and temperature conditions. After a two-day heavy load transport by the transport company Wimmer, the centrepiece of the instrument, a large six-ram multianvil press with a positioning and turning table had recently been delivered and installed as the first instrument of the new neutron guide hall east at FRM II (figs. 1-6). Since the position of the instrument was predetermined by the future beam course and the exact distance to the upstream instrument POWTEX, the 70 ton unit of press and positioning table had to be placed and adjusted with millimetre accuracy in the press pit. Press and positioning table were built by the small upper Franconian company Voggenreiter that is specialized in the construction of large multianvil facilities for scientific and industrial use.

The multianvil press has six independently controlled rams that have a combined pressing force of up to 23.5 MN (2400 tons). The force is transferred and focussed through a set of smaller secondary anvils to a cubic assembly that includes the sample and an internal heating furnace (fig. 7). With this 6-6 anvil geometry it is possible to subject a large volume of crystalline solid material or melt to pressure conditions of at least 15 GPa (150 bar) and temperatures above 2000 K for performing neutron scattering and radiography measurements. Ultimately, the press will reach 25 GPa. In addition to maintaining extreme conditions hydrostatically for extended periods of time, arbitrary anvil pairs can be pushed into or retracted from the sample assembly to achieve controlled deformation with varying strain rates of  $10^{-2}$  to  $10^{-6}$  s $^{-1}$ .

The positioning and turning table allows for optimal vertical and horizontal adjustment of the sample centre in the beam. While first neutrons are expected to reach the hall in 2014, operation of SAPHiR will commence immediately after setup starting with press testing and calibration followed by optimising the sample environment for later neutron measurements. Parallel, off-line scientific use is planned to start in 2013 with static and deformation studies at high pressure and temperature for material and Earth science.

SAPHiR will share a beam line with the instrument POWTEX and is situated ca. 11 m downstream. A neutron guide to bridge the gap has been de-

Fig. 4-6: SAPHiR arriving at the FRM II.

veloped with the Monte Carlo simulation program VITESS. The comparably small sample size and the necessary sample environment for maintaining extreme conditions require high neutron beam intensity and focussing on the sample cross-section for scattering experiments. Furthermore, high resolution neutron radiography requires a low divergence beam (high L/D ratio). These requirements were met by constructing an elliptic neutron guide with a supermirror coating with m-values of 1.5 to 4 to ensure maximum beam intensity at the sample cross-section. The required m-values along the guide axis were simulated with the backtracing method of Houben et al. (2012). For low beam-divergence applications a series of high precision neutron slits are included that can shade varying parts at the rear end of the guide, thereby allowing to choose between a range of L/D ratios as required. The neutron guide will be delivered and installed in the second half of 2013.

Time-of-flight neutron diffraction will be performed with neutron pulses of a wavelength range of 1 - 2.4 Å that are shaped by the POWTEX disc chopper cascade. Future applications of SAPHiR include the investigation of crystal structure and stability of high pressure phases, phase diagrams and transformation kinetics of light materials, and rheological flow laws at high-pressure and temperature. Neutron radiography will be performed with a low-divergence beam to allow for high resolution imaging and tomography. Applications include critical behaviour and miscibility of fluid-silicate melt systems, melt/fluid distribution in crystalline silicate systems, and the investigation of sintering kinetics. Especially the sensitivity of neutrons for light elements is very interesting for material science applications including the investigation of new super hard materials, as well as for Earth science by gaining a better understanding of the deep hydrogen and carbon cycles that are of immense importance for the evolution and dynamics of the Earth.

We are very happy that our instrument was successfully installed and are eager to start with first experiments. We would like to acknowledge the people who helped with this project including the leadership, scientists and support staff of FRM II, our colleagues from POWTEX, the construction company Voggenreiter, and the transport company Wimmer.

The project is funded by BMBF grant 05K10WC2.

*Nicolas Walte, Bayerisches Geoinstitut*

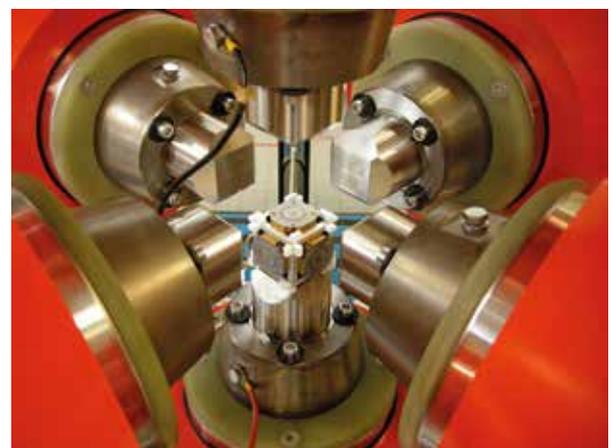


Fig. 7: Internal geometry of the press. Six primary rams compress a stack of secondary anvils guided in an aluminium frame that contains the sample assembly.

## Fatigue Process in Li-Ion Cells

### An in situ combined neutron diffraction and electrochemical study

In the last two decades the development of Li-ion batteries can be considered as the most remarkable success of modern electrochemistry leading to portable energy storage media possessing high energy density, operating at high voltages and showing good cycle life along with excellent storage characteristics. These facts make Li-ion batteries widely used in the field of portable electronics, but the technology progress constantly demands further developments, especially concerning enhanced rate capability, safety features, improved chemical/ structural stability of electrodes, capacity optimisation etc.

(LiCoO<sub>2</sub> cathode, graphite anode) were cycled up to 1000 times (3.0-4.2 V, CCCV, 1C) at 25 and 50°C. The effect of the battery fatigue on their electrochemical behaviour was successfully investigated. The influence of the cycling temperature appeared to be one of the major factors defining the battery performance.

The capacity loss determined from electrochemical investigation was found to be 21.9% and 12.8% for the battery cycled for 1000 times at 25°C and 50°C respectively, indicating a favourable influence of higher cycling temperatures on the battery performance (fig. 1).

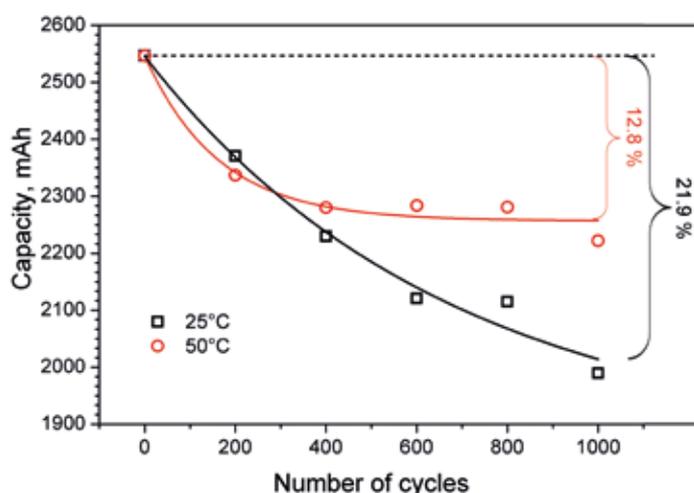


Fig. 1: Evolution of the measured cell capacity with cycle number.

Solid fundamental study of battery materials behaviour under real operation conditions of electrochemical cycling is needed for failure mechanisms identification, and further cell development. This type of “in situ” experiments on the industrial cells should be non-destructive, where all materials of the battery remain under real operation conditions and any risks of materials oxidation, electrolyte evaporation or battery charge change are eliminated. For this purpose the neutron scattering and especially the high-resolution neutron powder diffraction shows excellent performance due to advantages of this technique, i.e. the high penetration of thermal neutrons, the ability to localize light atoms (e.g. hydrogen, lithium) etc.

The commercial cylindrical 18650-type rechargeable Li-ion batteries

The Rietveld refinement technique was successfully applied for structural characterization of electrode materials during the fatigue process. The high-resolution neutron powder diffraction data were collected from the fully charged and discharged cells using the powder diffractometer SPODI. The diffraction pattern was simulated as a contribution of six phases corresponding to

- LiCoO<sub>2</sub> as a cathode material;
- Li intercalated carbons (anode material in charged state) and
- graphite (anode in discharged state of the cell);
- Cu and
- Al – both as materials from the current collectors and
- iron from the steel housing (fig. 2).

The evolution of structural changes was monitored as a function of battery fatigue, e.g. the Li occupations, change of lattice parameters and interatomic distances in structure of the Li<sub>x</sub>CoO<sub>2</sub>. Analysis of the acquired neutron diffraction patterns established the direct correlation and per-

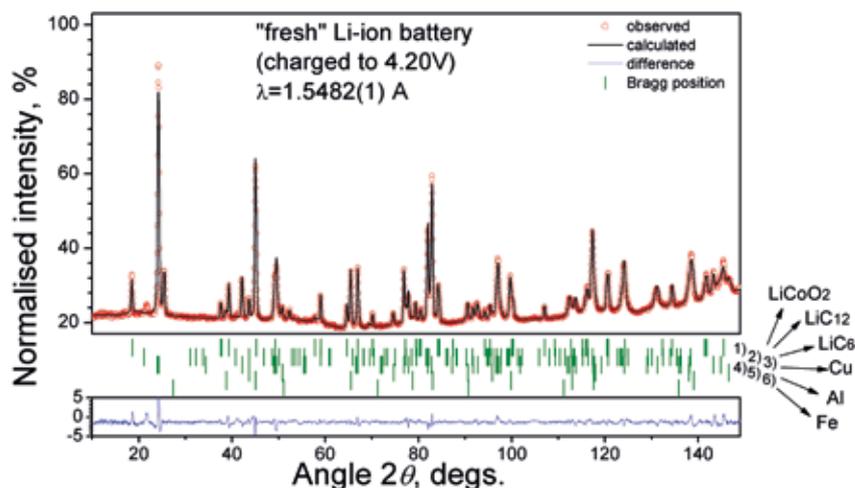


Fig. 2: Rietveld refinement of a neutron powder diffraction pattern acquired for a “fresh” battery charged to 4.20 V

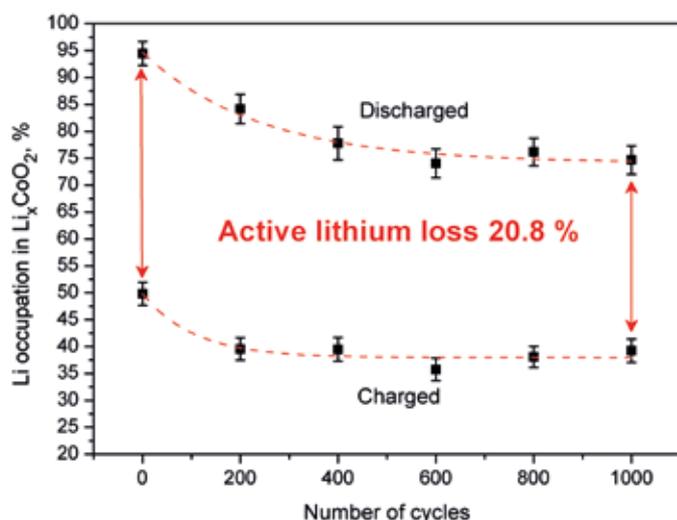


Fig. 3: Effect of battery fatigue at 25°C on the Li site occupation in  $\text{Li}_x\text{CoO}_2$ .

fect agreement between the capacity loss measured by means of electrochemistry and the Li occupation in the  $\text{Li}_x\text{CoO}_2$  (fig. 3). A reduction of an amount of exchangeable lithium by 21% and 12% was observed for the cell cycled at 25°C and 50°C, respectively, and shows a good agreement with electrochemical analysis.

Structural refinement of the anode exhibits formation of  $\text{LiC}_6$  and  $\text{LiC}_{12}$  intercalated compounds with different weight fraction depending on the level of the battery fatigue. The effect of cycling on the anode composition is shown in fig. 4. The changes of the weight fractions of intercalated phases correspond to a reduction of the available amount of active Li, which is inserted into the graphite during the charging. The quantitative recalculation of the intercalated Li into anode showed a reduction of 26% and 15% for the cells cycled at 25°C and 50°C, respectively. A slight increase of graphene interlayer spacings which could be related to the irreversible lithium or electrolyte trapping with battery cycling, was observed as well. Along with the structural changes the weak reduction of the crystallite sizes with cell degradation can be the additional reason for the capacity fade of a cell.

The loss of the active lithium in the cell is considered to be the primary reason for fatigue in Li-ion batteries. The most obvious scenario for the loss of mobile Li is its trapping in the formed solid electrolyte interface layer and

graphene interlayer. The successful results of the present study open us new perspectives for further investigations of commercial Li-ion batteries.

This work is financially supported by the Deutsche Forschungsgemeinschaft DFG within the transfer project *Operando studies of fatigue in commercial Li-ion batteries by neutron tomography and diffraction* as part of the Research Collaborative Centre 595 *Electrical fatigue in functional materials*.

Oleksandr Dolotko, FRM II

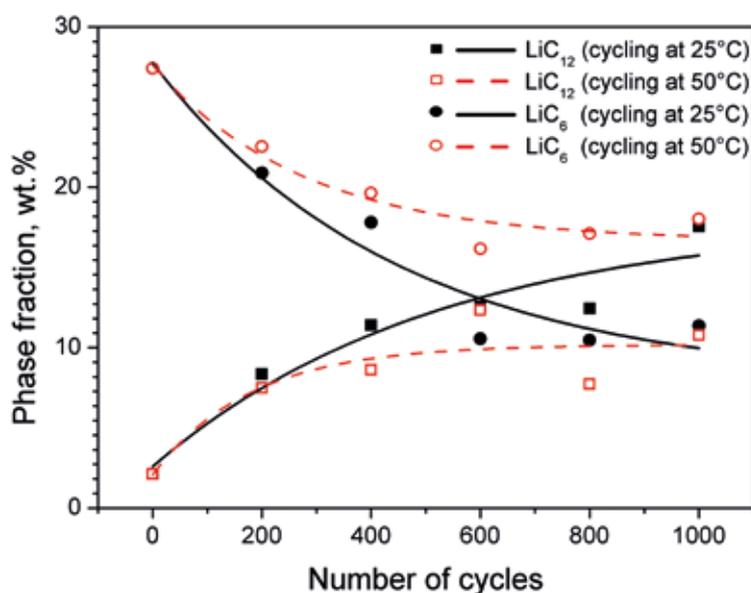


Fig. 4: Evolution of  $\text{LiC}_6$  and  $\text{LiC}_{12}$  phase fraction with cell fatigue.



## BMBF Grant for the TANDEM Collaboration

### Trans-uranium Actinide Nuclear Data – Evaluation and Measurement

Nuclear data are of great importance in nuclear research and industry. The accuracy of these data, mainly that of neutron capture cross sections affects the reliability of simulations and design of nuclear facilities from irradiation experiments through reactor-core calculations to transmutation. There are many compilations and databases available worldwide, however, the quality of these data is far from the users' requirements. The only way to improve the reliability of these values is to re-determine them experimentally.

Our main goal is to measure the thermal neutron capture cross-sections of trans-uranium actinides with high accuracy. Actinides are radioactive, thus the neutron-induced gamma radiation can be properly detected only in a high-flux neutron beam. The PGAA facility at Garching uses the strongest neutron beam in the world, which offers unique opportunities for this type of experiments. At the same time we are going to investigate the cross sections at fast neutron energies.

Activation measurements in cold neutron beams can be performed very precisely. First, the detector has to be calibrated (see fig. 1). This can be performed with high accuracy using standard calibration sources, whose decay schemes are known very well (see fig. 2). After that, the beam flux has to be determined using a gold foil or other comparator, the capture cross-section of which was determined with a high accuracy. Then a known amount of the investigated nuclide has to be irradiated in the beam, and from the detected areas of the characteristic peaks one can determine the activities and the partial cross sections for individual gamma lines. From the level-scheme information and the partial cross sections the total capture cross section can also be determined.

The colder the neutron beam, the higher the capture cross sections. In the cold energy range, every nuclide behaves regularly, i.e. all the cross sections as a function of the neutron energy change proportionally. Thus, when related to the thermal

cross section of the comparator, thermal cross section of the nuclide of interest will be determined in a cold beam.

The most accurate measurement can be performed, when the comparator nuclide is homogeneously distributed in the sample, because that takes into account the neutron self-shielding inside the sample. This can be achieved using e.g. stoichiometric compounds (like metal chlorides), or water solutions.

The trans-uranium actinides are important components of the nuclear waste. The reliability of their capture cross sections is of great importance in transmutation calculations and experiments. The available capture cross section data are highly unreliable (see table 1).

Some of these nuclides, as can be seen, have a half-life of more than a thousand years. That is why they remain in the nuclear waste in significant

amounts for a long time. These nuclides can also be activated in a neutron beam with a good signal-to-background ratio, as the contribution from natural activity is relatively small. At least 1 mg has to be irradiated from each material, but the lower cross section nuclides will need an at least an order of magnitude more. So far a demonstration experiment was performed in the medium-flux cold beam for  $^{237}\text{Np}$  and  $^{242}\text{Pu}$ .



Fig. 1: High-purity germanium detector with Compton-suppression system in an open lead shielding.

Forschungszentrum Jülich and FRM II at Garching applied for a grant of the Bundesministerium für Bildung und Forschung (BMBF) and the common project called *PGAA-Actinide* had been approved with a start in August 2012 and is running for three years (up to July 2015). The BMBF supports this project with 1.02 Mio€ (coordinator is Matthias Rossbach, Forschungszentrum Jülich), of which FRM II receives 420.000 €.

This financial support will be mostly used for building up a new low-background counting chamber close to the PGAA station, for the purchase of a

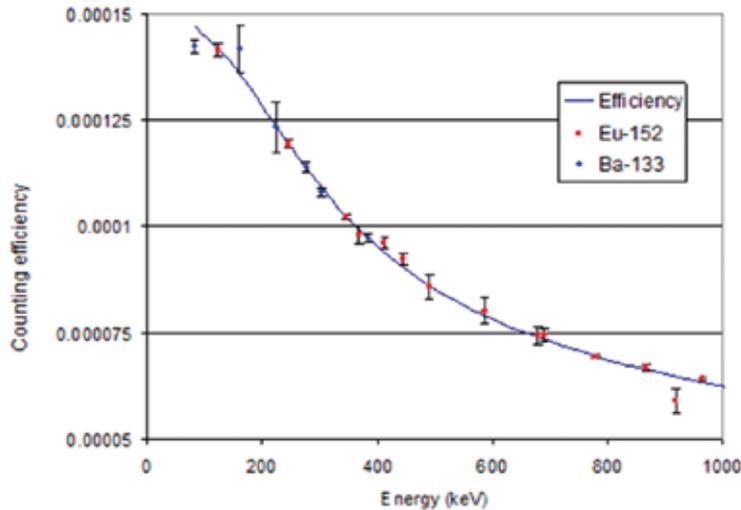


Fig. 2: The counting efficiency of the detector system at low energies. An 8<sup>th</sup>-order polynomial was fit to experimental data.

new high-resolution HPGe detector, and a three-year expert position at the PGAA facility, together with travel money for dissemination of the results and for meetings with the project partners (Jülich, Budapest). The FRM II part within the project is mostly concentrated on cold neutron PGAA experiments on the actinides of interest.

Additionally, the expertise and manpower for the installation works at NECTAR facility at FRM II will be given by the local PGAA team. We are going to install and test a fast-neutron PGAA instrument at the NECTAR facility in the frame of the BMBF project. The new instrument will enable irradiations with fast neutrons following the fission spectrum (from about 0.5 to 5 MeV). Reactions, other than neutron capture, like inelastic neutron scattering or charged particle emitting reactions will also be investigated. The data obtained in these measurements will be useful for irradiation experiments with neutron generators, too.

The collaboration was started as a German-German partnership, but it has rapidly grown to an international level:

- Matthias Rossbach, Christoph Genreith of Forschungszentrum Jülich, IEK-6: Safety Research and Reactor Technology
- Zsolt Révay, Petra Kudejova of Technische Universität München, Forschungs-Neutronenquelle Heinz Maier-Lebnitz (FRM II), PGAA
- Tamás Belgya of Institute for Energy Research of the Hungarian Academy of Sciences, Laboratory of Nuclear Analytics and Radiography
- Richard B. Fireston of Lawrence Berkeley National Laboratory, Isotopes Project

The partners in Budapest take part in the irradiations, while the Isotope Project from Berkeley is responsible for the level-scheme consistency, necessary to derive the capture cross section from the partial values. The Lawrence Livermore National Laboratory and the National Institute for Standards and Technology is also interested in the cooperation.

*Zsolt Révay, FRM II*

Nuclide	$\sigma_n$ (barn) ENDF	$\sigma_n$ (barn) JENDL	$\sigma_n$ (barn) JEFF	Half-life (year)	Specific activity (Bq/mg)
<sup>237</sup> Np	180	178.1	195.8	$2.144 \times 10^6$	$2.61 \times 10^4$
<sup>239</sup> Pu	289	271.5	272.4	$2.411 \times 10^4$	$2.30 \times 10^6$
<sup>241</sup> Pu	360	363	363	14.290	$3.84 \times 10^9$
<sup>242</sup> Pu	19	19.88	18.8	$3.73 \times 10^5$	$1.47 \times 10^5$
<sup>241</sup> Am	550	684.3	647	432.6	$1.27 \times 10^8$
<sup>243</sup> Am	78	79.26	76.7	$7.370 \times 10^3$	$7.39 \times 10^6$
<sup>243</sup> Cm	130	131.4	130.2	29.1	$1.87 \times 10^9$
<sup>244</sup> Cm	15	15.24	10.4	18.11	$3.00 \times 10^9$
<sup>245</sup> Cm	360	347	359.1	$8.5 \times 10^4$	$6.36 \times 10^5$

Table 1: Thermal neutron capture cross sections of a few trans-uranium actinides from different sources.

## Investigation of Scheelite-Type Multiferroics Minerals

### Using naturally occurring large single crystals with high-quality at RESI

In the recent years the interest in materials exhibiting both ferromagnetic and ferroelectric properties has strongly been increased. These so-called multiferroics displaying extremely complicated structural properties belong to one of the most interesting, application-oriented research objects in our time. For example, they are discussed as building blocks for spintronic devices where information is encoded in spin-states, used in sensitive magnet field sensors or in microwave applications. But currently most technical applications still use multiphase systems as there are only very few single phase compounds that exhibit multiferroic properties. Fundamental multiferroic states arise from charge ordering possible in diverse structure types, as being induced by

- ordering of transition metal cations with mixed valence;
- lattice distortion leading to polar symmetry and missing inversion;
- magnetic frustration.

As a consequence of this complexity only a limited number of multiferroics has been known, and details of their multiferroic properties are still to be explained.

The group of Sohyun Park at the Ludwig-Maximilians-Universität München is working on natural occurring

minerals as a base for different potential materials for industrial applications. Among the fields of interest are zeolithes as ion-conductors and scheelite-type minerals as potential multi-ferroic materials. The group is a member of the Sektion Kristallographie, Department für Geo- und Umweltwissenschaften under the guidance of Wolfgang Schmahl. The group uses lab-X-ray, synchrotron and neutrons as methods for a better understanding of structural properties and is one of the partners operating the single crystal diffrac-

tometer RESI at FRM II.

To gain a deeper insight into the multi-ferroic properties we have started to investigate scheelite ( $\text{CaWO}_4$ ) and willemite ( $\text{Zn}_2\text{SiO}_4$ )-type materials (so-called  $\text{AXO}_4$  groups). Fortunately many of them crystallize naturally as well-formed single crystals, such as hübnerite ( $\text{MnWO}_4$ ), ferberite ( $\text{FeWO}_4$ ), and wolframite ( $(\text{Fe, Mn})\text{WO}_4$ ). Centimetre-sized single crystals with a high quality could be provided by Mineralogische Staats-Sammlung

München.

In our preliminary work, we select single crystals from five materials of this class to collect synchrotron X-ray single crystal diffraction (XSD) data at the instrument F1, HASYLAB at DESY, Hamburg. The selected minerals are scheelite ( $\text{CaWO}_4$ ), hübnerite ( $\text{MnWO}_4$ ), stolzite ( $\text{PbWO}_4$ ), wolframite ( $(\text{Fe, Mn})\text{WO}_4$ ), and wulfenite ( $\text{PbMoO}_4$ ). These compounds crystallize in centrosymmetric space groups at room temperature (RT) but at lower temperatures it is expected to observe structural changes leading to multiferroic couplings such as lattice distortion, charge ordering, loss of inversion centre or spiral magnetic ordering. Among the selected minerals, only hübnerite ( $\text{MnWO}_4$ ) was structurally investigated



Fig. 1: RESI

in 1969. Multi-ferroic properties in hübnerite have been reported based on neutron single crystal and powder diffraction data collected on different instruments at various phase transition temperatures. To study the entire structural properties in the superspace in detail we intend to effectively collect a high amount of superstructure reflections subsequently before and after phase transitions. Our SXD data show no distinct phase transition due to nuclear structural changes from 293 down to 100 K. In addition there are problems due to the

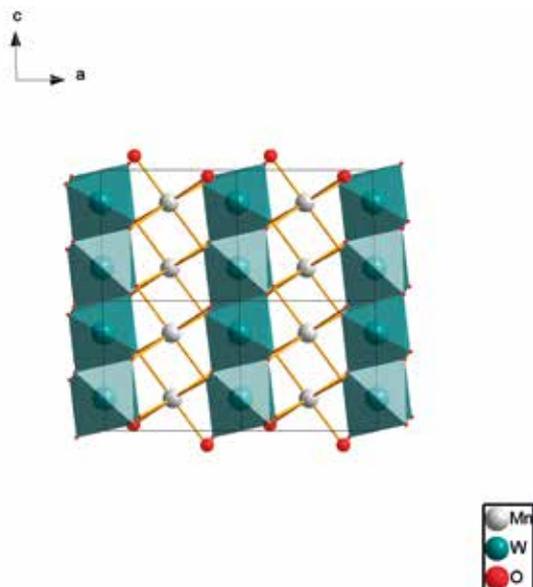


Fig. 2: Atomic structure of hübnerite seen along the b-axis. One can clearly see chains of  $WO_4$ -octahedron and Mn-chains.

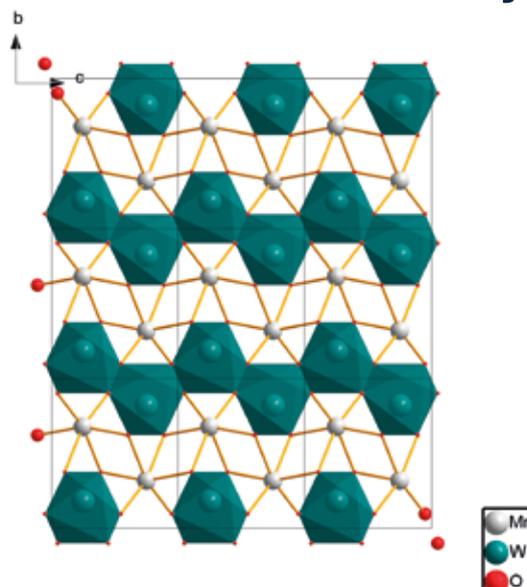


Fig. 3: Atomic structure of hübnerite seen along the a-axis. In this direction Mn forms zig-zag chains.

high absorption in XSD with our W- and Pb-compounds. The use of neutron radiation is therefore essential to monitor phase transitions for possible ferromagnetic ordering. It is crucial to conduct neutron single crystal diffraction in a wide range of temperatures to find a good performing multiferroic system working at high temperatures. As many multiferroic systems are forming non-commensurate phases (both structurally and magnetically), an area detector is essential to be able to observe the corresponding diffraction patterns easily. The instrument RESI with its wide range of temperatures available and the high-resolution image plate detector is therefore a natural choice. A systematic neutron single crystal diffraction study with diverse compositions of  $AXO_4$  group members will help to further understand multiferroic mechanism in the selected system. For this purpose large single crystals with several mm edge lengths from the same batches as taken for the XSD study were selected and will be used for

the neutron experiments. To determine chemical composition data acquisition using electron microprobe is currently in progress. The main work will be done by a new Ph.D student in the group of Sohyun Park, David Behal.

The selection of suitable crystals mainly from the large mineralogical state collection will be continued on other similar crystal families as well.

In October, we collected three complete data sets at 3 K, 7 K, and 13 K with a hübnerite single crystal ( $3.3 \times 6.6 \times 17.7 \text{ mm}^3$ ) at the instrument RESI. Results from data reduction are summarised in table 1, showing commensurate modulation at 3 K and incommensurate modulation at 7 K and 13 K in agreement with those reported in the literature. Details of structure analysis with the current neutron data sets in combination with studies of domains of hübnerite using Raman spectroscopy and synchrotron single crystal diffraction will be reported elsewhere.

Sohyun Park, LMU  
Bjørn Pedersen, FRM II

Temperature	3 K	7 K	13 K
Lattice parameter	$a = 4.816 \text{ \AA};$ $b = 5.743 \text{ \AA};$ $c = 4.979 \text{ \AA};$ $\beta = 91.05^\circ$	$a = 4.810 \text{ \AA};$ $b = 5.742 \text{ \AA};$ $c = 4.975 \text{ \AA};$ $\beta = 91.02^\circ$	$a = 4.808 \text{ \AA};$ $b = 5.742 \text{ \AA};$ $c = 4.979 \text{ \AA};$ $\beta = 91.03^\circ$
q-vector	(0.25, 0.5, 0.5)	(0.216, 0.5, 0.535)	(-0.215, 0.5, 0.456)
All and unique reflection numbers	2911; 1440	1776; 667	1775; 666
$R_{\text{ini}}$ for main reflections with $I > 3\sigma(I)$	4.04	5.07	5.35

Table 1: Data reduction of single crystal neutron diffraction data of hübnerite, collected at RESI, FRMII.

## Newly Arrived

I'm second instrument scientist in charge of the high intensity small angle scattering diffractometer KWS-1 of JCNS and I will be responsible for implementation of polarized neutrons and polarization analysis at the instrument. I finished my PhD in Taras Shevchenko Kyiv National University (Ukraine) by performing experiments at Frank Laboratory of Neutron Physics of Joint Institute for Nuclear Research in Dubna (Russia). I'm interested in structural studies in condensed matter physics.



### KWS-1

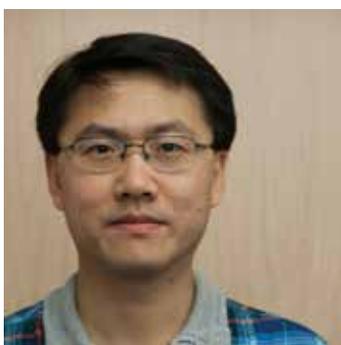
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I am an instrument scientist at the very small angle scattering diffractometer KWS-3 of JCNS. Before joining the team at Garching, I finished my PhD at Forschungszentrum Jülich on molecular and molecule-based magnets. My scientific interests are nanoscale magnets, spin-frustrated magnets and magnetic-caloric effect in molecule-based magnets.

I am a new JCNS postdoc investigating the dynamics of microgels and guest particles on J-NSE instrument. I've got my PhD in Physical Chemistry at the Westfälische Wilhelms-Universität Münster carrying out a SANS study on polymer modified microemulsions at Forschungszentrum Jülich. After that, I worked as postdoc at Université de Montpellier 2 (France) and University of Massachusetts Amherst (USA). My field of interest is soft condensed matter (self-assembly of polymers, surfactants and proteins in solutions).



### J-NSE

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### DNS

#### Kirill Nemkovski

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I am the new second instrument scientist at DNS spectrometer operated by JCNS. Before I was the second instrument scientist at the new thermal time-of-flight spectrometer with polarization analysis TOPAS that is constructing by JCNS in the neutron guide hall east. My research interests are related to magnetic excitations and lattice dynamics in strongly correlated rare earth intermetallics (heavy-fermion and mixed valence compounds, Kondo insulators), frustrated transition metal oxides and systems with large magneto-caloric effect.

## Newly Arrived

### PUMA

#### Jitae Park

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I am instrument scientist at the thermal neutron three-axes spectrometer PUMA. Before I joined FRM II, I completed my PhD thesis at the MPI for Solid State Research (Stuttgart). It was mainly related to the magnetic excitations in iron-based superconductors, and PUMA was one of the most frequently used instrument for my PhD work. I am especially interested in high-Tc superconductivity, strongly correlated electron system, itinerant magnetism in intermetallic system, various ordering phenomena, as well as the cutting edge instrumentation development.

I am the Information Manager of NMI3, the European Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy. I am responsible for updating the NMI3 website and for communicating the NMI3 activities, events and achievements to the scientific community and the public through the newsletter and website. I have studied environmental engineering. Previously to the NMI3 I have worked for 4 years at the Joint Research Centre of the European Commission carrying out research on science communication and public reception of media. I believe that it is fundamental to communicate science in order to reach out to the public, policy-makers and scientific community. This position is the perfect opportunity to do so!



### NMI3

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*A  
Merry  
Christmas  
and  
a  
Happy  
New Year  
to all  
our users!*

## From North to South: GEMS at FRM II

### The German Engineering Materials Science Centre Outstation



Nearly the whole GEMS-team at the FRM II:

Martin Haese-Seiller,  
Armin Kriele,  
Sven-Arne Siegfried,  
Weimin Gan,  
André Heinemann,  
Christian Schwarz,  
Matthias Pomm,  
Jean-François Moulin,  
Jörgen Francke  
(from left to right).

The Helmholtz-Zentrum Geesthacht (HZG, formerly GKSS) has started activities at the FRM II in 1998, when it began building the horizontal TOF reflectometer REFSANS. Since then the involvement of HZG in Garching has been growing and shared operation of the residual stress and texture diffractometer STRESS-SPEC was initiated together with FRM II, followed by the joint project of the large SANS-1 instrument. The operation of the three instruments has then been placed under the supervision of the German Engineering Materials Science Centre (GEMS) which was created by HZG to serve as a central user access platform to its instruments at large scale facilities (synchrotron at Desy and neutron beamlines at FRM II).

This family of three instruments is emblematic of the activities of GEMS which span fields ranging from engineering materials over soft-matter to magnetism, observed on a very broad range of organisation scales and bridging fundamental research with direct industrial application. Beside these existing instruments HZG is also planning to increase its involvement at FRM II by filling a new research position in the field of neutron tomography.

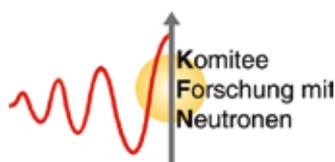
Making this happen is of course only possible with a team. The local GEMS group in Garching is composed of Jean-François Moulin (head of the outstation and responsible for REFSANS), André Heinemann (second instrument scientist at SANS-1), Weimin Gan (instrument scientist in charge of the texture measurements at STRESS-SPEC), Heinz-Günter Brokmeier (HZG/TU Clausthal, robot pro-

ject at STRESS-SPEC), Helmut Eckerlebe (local contact at SANS-1), Martin Haese-Seiller (beamline scientist at REFSANS), Matthias Pomm (instrument engineer at REFSANS), Jörgen Francke (mechanical designer, supporting all three instruments), Armin Kriele (responsible for the Hard-Soft-Matter Lab, a facility also presented in this issue of FRM II news), Svatopluk Semecky (technician, partly funded), Sven-Arne Siegfried (PhD student at SANS-1/REFSANS), Nowfal Alhamdany (PhD student at STRESS-SPEC) and Christian Schwarz (who joined the REFSANS group for an internship).

Beside the daily life of any beamline, some of the ongoing projects of the group are:

- at SANS-1: To bring it into user operation and implement its dedicated sample environments.
- at REFSANS: To provide a high quality polarised beam useable for reflectometry and GISANS, which will be used not only for magnetism studies but also to reduce the incoherent background contribution from high H content samples typical of soft matter.
- at STRESS-SPEC: To improve the implementation of the already existing robotic sample handling system and its accuracy in order to perform stress or local textures measurements on bulky and/or complex shaped samples.
- at the Hard-Soft-Matter Lab: To provide a good technical support to the user community and broaden the spectrum of available preparation/characterization methods.

*Jean-François Moulin, HZG-GEMS*



## Strengthening of neutron research in Germany and Europe

### Preparing the way for new innovations

Not only for the KFN it was a busy fading of the year 2012. Also many of the German neutron research groups have been very active in writing their applications for projects within the new round of “Verbundforschung”. It was a great pleasure for me to realize a broad activity throughout the whole community in the last months of the year and I am convinced that a large number of very competitive proposals will be finalized and submitted in time before the deadline on the 10<sup>th</sup> of December. This impressively demonstrates that the large and well-known community of neutron research groups at German universities does not only use neutron facilities for their daily research but is also strongly involved in neutron instrumentation. In well established or new created cooperations with the large neutron centres the expertise and the cost efficient and research driven working methods of the groups significantly contribute to the generation of new and innovative instrument solutions.

Not only the broad scientific activity of the German users’ community but also of our invited colleagues from Sweden, Denmark, the Netherlands, and Belgium was demonstrated on the *German Conference on Neutron Scattering 2012* in Bonn. The impressive number of young scientists attending the conference and the very competitive young researchers proposed as candidates for the Wolfram-Prandl-Prize 2012 convinced me that our community is well-prepared for the future as it creates a broad new generation of excellent young academics enthusiastic for our research. I congratulate Dmytro Inosov as the winner of the Wolfram-Prandl-Prize 2012. I would like to thank the Forschungszentrum Jülich very much for the perfect organisation of the conference which was the basis of its big success.

Besides such particular events it was a continuous work in 2012 to contribute to the development of neutron facilities in Germany and Europe. The main project in this respect is certainly the upcoming European Spallation Source (ESS). The extending enthusiastic support of the ESS project all over Europe is highly encouraging. Nevertheless, there are ongoing discussions on how to handle the contributions of the different countries and how to organise and synchronize the many different cooperations. At the same time the KFN strongly expresses the need to further strengthen the existing German neutron sources as they are the essential basis to preserve sustainability and excellence of German neutron research.

The KFN is strongly involved in these discussions and tries to express a representative opinion of the German users’ community. Therefore, we need to stay in close contact with the community. Thus, I kindly ask every neutron user to communicate to us her or his opinion and/or concerns about the near and further future of neutron research in Germany and Europe. This will help us to convincingly represent the viewpoint of the users’ community.

Finally, I would like to thank the many people who supported our work in the last year. I wish all of you a Merry Christmas, a relaxed finish of 2012, and a Happy New Year. I am looking forward to seeing you in 2013 and continuing our work for neutron research in Germany.

Tobias Unruh  
Chairman of the 9<sup>th</sup> Komitee Forschung mit Neutronen (KFN)  
[Tobias.Unruh@fau.de](mailto:Tobias.Unruh@fau.de)



## First Joint Review Panels Meeting!

Ismaning, September 13<sup>th</sup>-14<sup>th</sup>



It was not only the first joint meeting, it was also the first two-day meeting, it was the first time ever for several participants and finally: It was the first meeting held at a conference centre in Ismaning instead of the FRM II site as always.

After taking some refreshments, the reviewers were welcomed by the scientific directors, Winfried Petry and Dieter Richter, and Flavio Carsughi gave a short overview of news from the instruments. Following this, the panels started their discussions which lasted into the evening. Then it was time for a short walk: The dinner was served in the Gasthof zur Mühle and the participants not only enjoyed the Bavarian meal but also used the time for discussions between the individ-

ual panels. The following morning was devoted to the work of the panels' again.

During the final meeting of all panels' chair persons, secretaries, scientific directors, and the User Office in the afternoon of September 14<sup>th</sup>, a very positive feedback was given. The amalgamation of the former separated panels of FRM II and JCNS was felt as a very successful step forward. The chair persons pointed out that due to

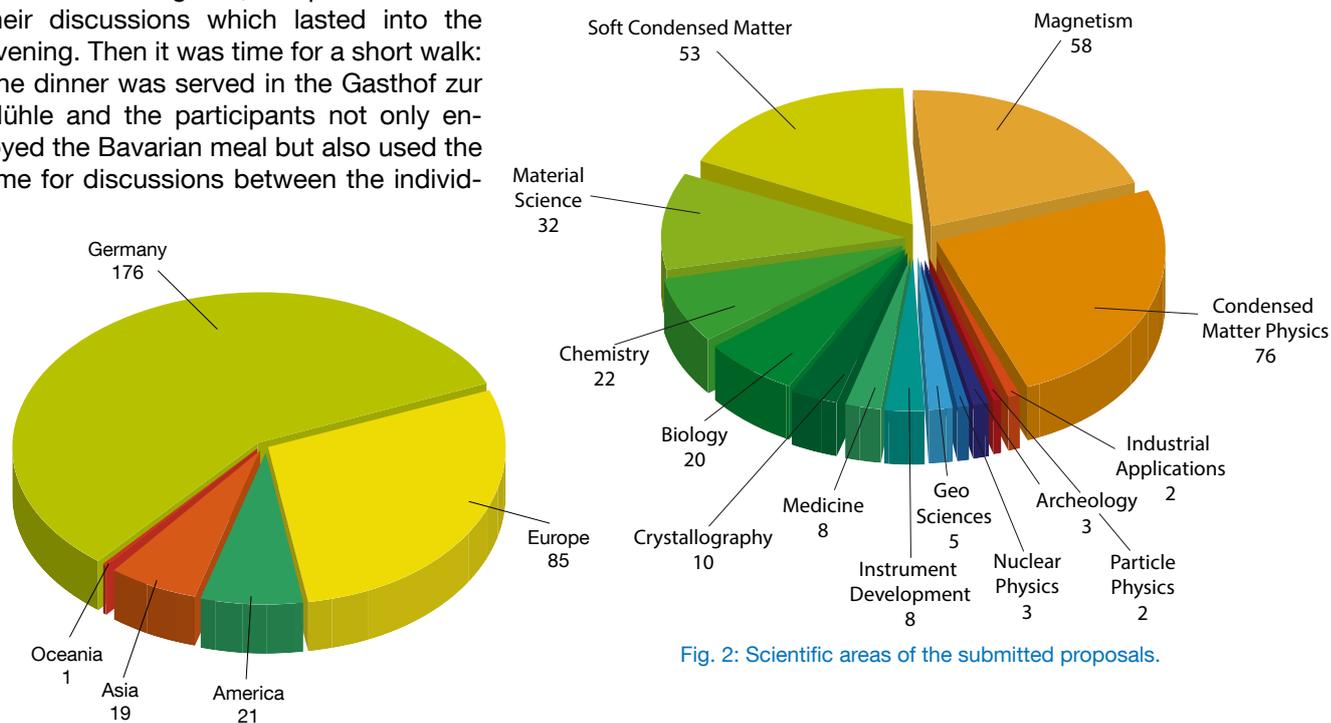


Fig. 1: The main proposers' countries.

Fig. 2: Scientific areas of the submitted proposals.



The referees listening to Flavio Carsughi.

this reform, the work had gained more efficiency and the discussions had been very concentrated and therefore fruitful. The physical proximity had opened up the possibility of quick exchanges of ideas between the panels - and this was very appreciated.

All proposals submitted to the instruments operated by Forschungszentrum Jülich, HZG, HZB, MPG and TUM at the FRM II were discussed within this professional setting. Furthermore, the panels had also to deal with proposals requesting CRG beam time at JCMS instruments at SNS and ILL. The selection process was very tough and the work of the 56 reviewers was not an easy one. The quality of the submitted proposals was in general quite high, but unfortunately we could not accept all as the global overload factor was about 2.0.

In total the User Office received 302 proposals, requesting 2.038 beam days of which 1.104 could be allocated. The number of proposals per review panel ranges from 28 (*Biology*) to 79 (*Magnetism and Spectroscopy*). This difference was partly compensated by the size of the review panels.

The powder diffractometer SPODI was very popular among the users: The most



Lively discussion during the dinner.

proposals were submitted to this instrument: 29. However, the most days were requested at PUMA. The proposers asked for 146 days of beam time at the three-axes spectrometer.

The main proposers worked predominantly in Germany (58%) and in other European countries (28%). The rest was shared between America, Asia and Oceania (fig. 1).

The scientific area (this assignment is done by the proposers themselves) which received the largest number of proposals was that of condensed matter physics. It got 76 proposals. Fig. 2 illustrates that more than 86% of the submitted proposals were related to six out of the 14 available scientific areas.

The number of submitted proposals in the year 2012 was similar to that of 2010 (fig. 3). Due to the long maintenance break in 2011, only one proposal round could be held then. We are happy to see that the users' interest in the instruments at the FRM II have not fallen - and we look forward to receiving even more proposals for the next round. So don't miss the deadline on January 25<sup>th</sup>, 2013!

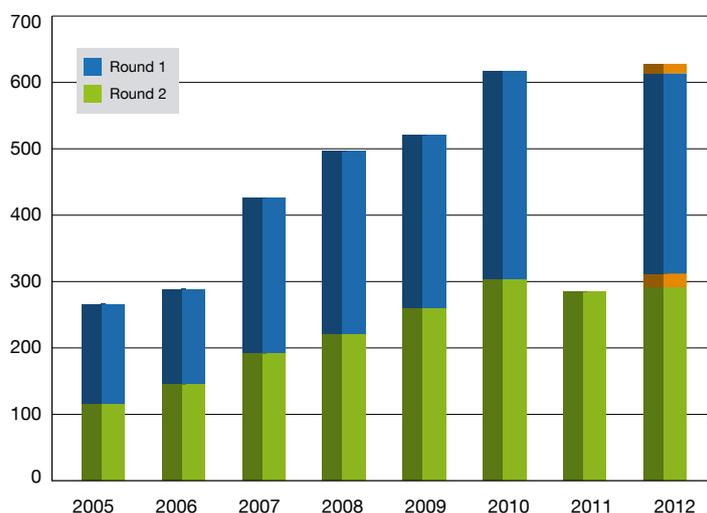


Fig. 3: Evolution of the submitted proposals. In 2012 also proposals submitted to the JCMS instruments at ILL and SNS are shown (orange).

## Sample Environment User Survey 2012:

[www.frm2.tum.de/en/user-office/user-survey-sampleenvironment](http://www.frm2.tum.de/en/user-office/user-survey-sampleenvironment)

At the FRM II the satisfaction of our users is of primary importance. Therefore, in February 2012, a user survey on the sample environment was launched and it will run until the end of the year. This user survey aims at the improvement of the service for the users as well as to identify the wishes for missing techniques in order to enlarge our offer.

For the time being, we received feedback on 20 instruments and 6 different sample environments (temperature, pressure, magnetic field, gas atmosphere, rheology stress and electric field) and the overall satisfaction of the users lies between good and very good in all the cases. Nevertheless, the users provided us with very useful general and specific comments, which will be very deeply analysed by our technicians and scientists.

Every user who performed a measurement at any instrument of FRM II is kindly invited to proceed to the web page given above and share her/ his experience on the sample environment with us.

The user survey consists of a general part with questions to all the users regardless of the sample environment unit used during the measurements, and of the experiment related part which deals with the units used during the measurements. The two parts are fully independent from each other and the second part shall be submitted for each combination of the instrument and sample environment used during the measurements. It is worth to remind you that the survey is fully anonymous.

We are working for you, and therefore, for your own interest, please take a few minutes to complete the user survey.

*Dear users,*

you are invited to apply for beam time at the German neutron source Heinz Maier-Leibnitz (FRM II).

**Deadline for proposals: January 25<sup>th</sup>, 2013**

Just **register** at the digital user office. With your personal account you can access the proposal and reporting system. Have a look at

[www.frm2.tum.de/en/user-office](http://www.frm2.tum.de/en/user-office)

for additional information and guidance to perform experiments at the FRM II.

### **Please note:**

Proposals have to be submitted via the web portals within your personal account

- for FRM II, HZG, HZB, MPG instruments: [user.frm2.tum.de](http://user.frm2.tum.de)
- for JCNS instruments: [fzj.frm2.tum.de](http://fzj.frm2.tum.de)

They are **reviewed** twice a year. The next joint **review** will take place on March 21<sup>st</sup>-22<sup>nd</sup>, 2013. Results of the review panel meeting will be online about two weeks later.

The FRM II is a partner in the EU supported network of European neutron facilities (**NMI3-II** in FP7). Researchers working in EU Member States or Associated States other than Germany can **apply for travel and subsistence reimbursement**:

[www.frm2.tum.de/en/user-office/nmi-3](http://www.frm2.tum.de/en/user-office/nmi-3)

**Researchers working at German universities** can apply for travel and subsistence reimbursement granted by the FRM II, JCNS, HZB and HZG:

[www.frm2.tum.de/en/user-office/financial-support](http://www.frm2.tum.de/en/user-office/financial-support)

To ensure the feasibility of the proposed experiment please contact the instrument scientist in advance.

Furthermore you can apply for CRG beam time at JCNS instruments at ILL and SNS for German users. For more information about this please refer to

[http://www.fz-juelich.de/jcns/DE/Leistungen/Userinfos3/\\_node.html](http://www.fz-juelich.de/jcns/DE/Leistungen/Userinfos3/_node.html)

In addition to beam tube experiments, irradiation facilities are available for neutron activation analysis, isotope production and silicon doping.

# Call for proposals: Next deadline January 25<sup>th</sup>, 2013



[user.frm2.tum.de](http://user.frm2.tum.de)

## Diffraction

### BIODIFF

diffractometer for large unit cells; cold source

### MIRA

multi purpose diffractometer; cold source

### RESI

single crystal diffractometer; thermal source

### SANS-1

small angle scattering instrument; cold source

### SPODI

powder diffractometer; thermal source

### STRESS-SPEC

material-science diffractometer; thermal source

## Radiography

### ANTARES

radiography and tomography; cold neutrons

### NECTAR

radiography and tomography;

fission neutron source

### PGAA

prompt gamma-activation analysis; cold source

## Spectroscopy

### PUMA

three-axes spectrometer; thermal source

### RESEDA

resonance spin-echo spectrometer; cold source

### TOFTOF

time-of-flight spectrometer; cold source

### TRISP

three-axes spectrometer with spin-echo; thermal source

## Reflectometry

### NREX

polarized neutron reflectometer; cold source

### REFSANS

time-of-flight reflectometer; cold source

## Positrons

### NEPOMUC

- positron beam (open beam port)
- positron defect spectrometer (Coincidence doppler broadening)
- positron life time spectroscopy (PLEPS)



[fzj.frm2.tum.de](http://fzj.frm2.tum.de)

## Diffraction

### BIODIFF

diffractometer for large unit cells; cold source

### HEIDI

single crystal diffractometer; hot source

### KWS-1

high intensity small angle scattering diffractometer; cold source

### KWS-2

small angle scattering diffractometer; cold source

### KWS-3

very small angle scattering diffractometer; cold source

### POLI

polarized hot neutron diffractometer; hot source

## Spectroscopy

### DNS

polarized diffuse neutron scattering; cold source

### J-NSE

neutron spin-echo spectrometer; cold source

### PANDA

three-axes spectrometer; cold source

### SPHERES

back-scattering spectrometer; cold source

## Reflectometry

### MARIA

magnetic reflectometer with high incident angle; cold source



## Upcoming

### February 26-28, 2013

24. SAAGAS - Seminar Aktivierungsanalyse und  
Gammaspektroskopie  
(Garching, Germany)  
[www.frm2.tum.de/saagas24](http://www.frm2.tum.de/saagas24)

### February 25 - March 8, 2013

44<sup>th</sup> IFF Spring School: *Quantum Information Science*  
(Jülich, Germany)  
[www.fz-juelich.de/SharedDocs/Termine/PGI/EN/2013/2013-02-25IFFSpringSchool44th.html](http://www.fz-juelich.de/SharedDocs/Termine/PGI/EN/2013/2013-02-25IFFSpringSchool44th.html)

### March 10-15, 2013

DPG Spring Meeting of the Condensed Matter  
Section  
(Regensburg, Germany)  
[regensburg13.dpg-tagungen.de/index.html?lang=en&](http://regensburg13.dpg-tagungen.de/index.html?lang=en&)

*Visit our booth there!*

### July 08-12, 2013

International Conference on Neutron Scattering  
(Edinburgh, United Kingdom)  
[www.icns2013.org](http://www.icns2013.org)

*Visit our booth there!*

### September 02-13, 2012

17<sup>th</sup> JCNS Laboratory Course - Neutron Scattering  
(Jülich/ Garching, Germany)  
[www.neutronlab.de](http://www.neutronlab.de)

### September 09-12, 2013

NINMACH 2013 - 1<sup>st</sup> International Conference on  
Neutron Imaging and Neutron Methods in Archaeology  
and Cultural Heritage Research  
(Garching, Germany)  
[www.frm2.tum.de/NINMACH2013](http://www.frm2.tum.de/NINMACH2013)

### September 15-20, 2013

13<sup>th</sup> International Workshop on Slow Positron  
Beam Techniques and Applications  
(Garching, Germany)  
[www.slopos13.com](http://www.slopos13.com)

### October 07-10, 2012

JCNS Workshop 2013: *Trends and Perspectives  
in Neutron Scattering: Magnetism and Correlated  
Electron Systems*  
(Tutzing, Germany)  
[www.fz-juelich.de/jcns/JCNS-Workshop2013](http://www.fz-juelich.de/jcns/JCNS-Workshop2013)

## IMPRINT

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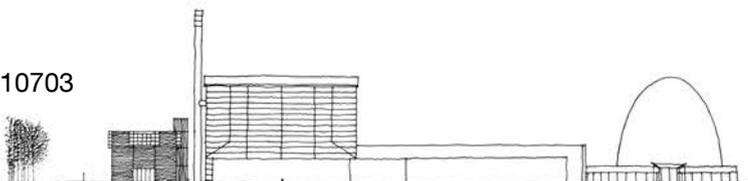
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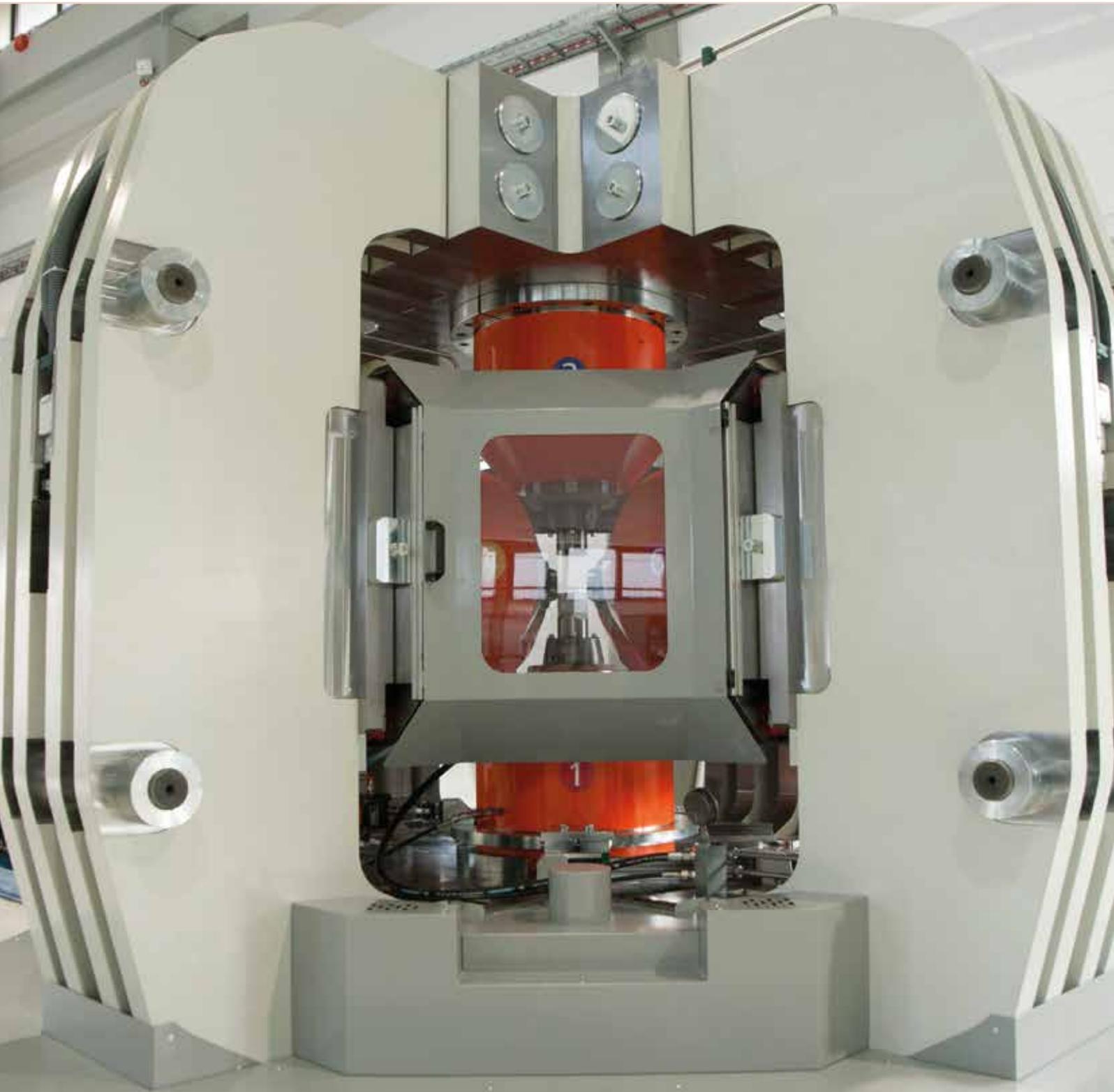


## *A Hard Day's Work....*

The construction work for the basement of the future connecting building between the Experimental Hall of the reactor building (to the right) and the Neutron Guide Hall East (to the left) started on June 24<sup>th</sup> and was finished within two months. It marks a milestone on our way to the future usage of the Neutron Guide Hall East.



news 9 (2012)



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