

### Fruitful and future cooperation

When the research reactor DIDO, at the Forschungszentrum Jülich (FZJ), was closed in May 2006, it was a logical step to join the world famous expertise and knowledge of neutron scattering from Jülich, with the equally well established research excellence in neutron science at the FRM II. The Jülich Centre for Neutron Scattering was subsequently established, with a massive investment in instrumentation and a build up of scientific and technical personal; giving rise to a second driving force at the FRM II. To date there are seven neutron scattering instruments either in the commissioning or operating phase at the FRM II. These include the innovative small angle scattering instrument KWS-3 that uses optical mirrors as focusing elements, the well known neutron spin echo instrument J-NSE and the backscattering instrument SPHERES with phase space transformation. The magnetic reflectometer MARIA that allows two dimensional polarization analysis is in commissioning.

The inclusion of the guide hall east into the atomic license means that future JCNS instruments are on the horizon. These include TOPAS (a polarized neutron time of flight spectrometer) and POWTEX (a high intensity time of flight diffractometer of RWTH Aachen and Universitiy of Göttingen constructed in collaboration with JCNS). In the near future a joint office building is planned by the FZJ and TUM and construction should begin soon. Aside from instrument development and operating a user service, Jülich offers a high level of scientific advice based on its strong research in nanomagnetism and soft matter. This is accompanied by specialized user laboratories for chemistry, biophysics and surface science.

The fruitful collaboration between JCNS and TUM will be strengthened in the near future by additional federal funding; this is governed by a cooperation agreement that also includes contributions from Helmholtz Center Berlin and GKSS in Geesthacht. Thus the future outlook for excellent research at the FRM II research reactor is bright and we look forward to further major scientific achievements. Deadline Proposal Rounds FRM II (N° 12) user.frm2.tum.de

**October 8th**, 2010

JCNS (N° 8) fzj.frm2.tum.de

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# **MIRA2: Extended possibilities**



Fig. 1: MIRA2 at the FRM II.

MIRA2 is the new beamport for the instrument MIRA providing an extended range of wavelengths; in particular the shorter wavelengths in the range 3.5 Å  $< \lambda < 6$  Å are now available. The wavelength is selected by a vertically focusing pyrolytic graphite monochromator, mounted within shielding on the neutron guide, NL6 in front of the DNS spectrometer. The new monochromator allows the use of the whole beam profile (60 mm x 120 mm) of the NL6 guide. In addition to having access to atomic resolution, the additional wavelength range is closer to the maximum of the neutron spectrum resulting in MIRA2 having a considerably higher flux of about 5.107 cm<sup>2</sup>s. The existing sample and detector units from MIRA can easily be attached to the MIRA2 port without changing sample environment, allowing the rapid realization of very flexible experiments.

Important applications of MIRA 2 will be:

- Cold neutron diffraction for the determination of magnetic structures. In particular large scale structures, eg. helical spin density waves or exotic magnetic lattices.
- Determination of structure and dynamics in extreme environments such as high pressure (currently being implemented). This option will utilize focusing guides before and after the sample and will allow a significantly higher flux for small samples.
- Determination of the layer thickness of films, for instance in polymer physics.
- Reflectometry from magnetic multilayers.
- Polarization analysis this is currently being installed.

First tests for wavelength calibration using  $\text{FeSr}_2O_{12}$  powder that provides a large d-spacing of 50 Å confirmed the expected wavelength range. The first user experiments have already been performed: Thomas Gutberlet (JCNS) and Mitch Croatt (ETH Zürich) studied the orientation and interaction of Amphotericin B (AmB) in phospholipid model membranes. AmB is a well-established antibiotic applied in the treatment of deep-seated mycotic infections. The incorporation of amphiphilic molecules of AmB into the lipid phase and their interaction with the lipid molecules leads to the destruction of the naturally selective membrane permeability. The AmB molecule appears to aggregate in the membrane in the presence of sterols thus forming pores, which kill the cell. The mode of action of AmB is based upon interactions with biomembranes. However, the exact binding properties of the antibiotic to the lipid membranes still remain obscure.

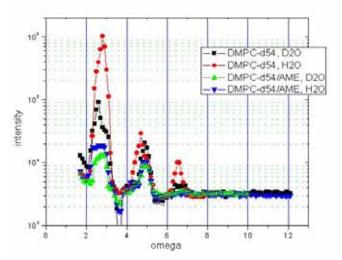


Fig. 2: Neutron diffraction profiles of oriented DMPC samples.

To study the interactions, Gutberlet and Croatt used oriented multilamellar stacks of dimyristoylphosphatidylcholine (DMPC) bilayers mixed with protonated and selectively deuterated AmB.  $\Theta/2\Theta$  diffraction experiments were performed using contrast variation. The obtained diffraction patterns of chain deuterated DMPC samples with or without AmB in H<sub>2</sub>O and D<sub>2</sub>O vapour are shown in figure 2. Up to four diffraction peaks could be recorded. Of these diffraction peaks Fourier coefficients to calculate neutron scattering length density (SLD) profiles were derived. The obtained neutron SLD indicated the presence of AmB in the centre of the hydrophobic bilayer core giving first hints on the binding properties of AmB.

In conclusion, MIRA2 has been successfully tested and will now be ready for user operation. It is now available for general users.

> Robert Georgii FRM II



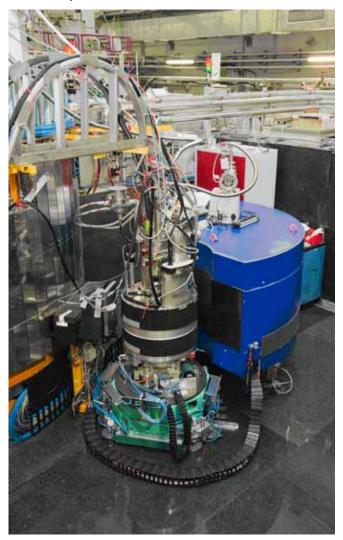
# 100 mK @ 7.5 T – extreme conditions at the push of a button

The sample environment group of FRM II marked another milestone in their ongoing effort to provide state of the art liquid-refrigerant free devices. A recent experiment at the three axes spectrometer PUMA used a combination of liquid-refrigerant free 7.5 T vertical magnet as well as a liquid-refrigerant free <sup>3</sup>He-<sup>4</sup>He dilution cryostat for the first time. The successful user experiment probed the behaviour in the ground state of the pyrochlore oxide spin-ice compound Dy Ti<sub>2</sub>O<sub>2</sub>. The ground state of this compound is interpreted as analog of the magnetic monopole. The experiment studied how the ground state evolves with temperature and field as one approaches the liquid-gas type critical point at (T $_{\rm c}$  = 0.4 K, H $_{\rm c}$  = 0.9 T) for the transition from low-density monopoles to high-density monopoles. (to learn more about "magnetic monopoles" see for example H. Kadowaki et al., J. Phys. Soc. Japan 78 (2009) 103706)

The sample environment set up which came into operation was a combination of the 7.5 T vertical field magnet, a top loading cryostat (CCR) and a dilution insert. Sample temperatures (in beam) were between 100-500 mK and magnetic fields up to 2 T were applied. All systems are liquid-refrigerant free, i.e. without liquid helium or nitrogen.

The liquid-refrigerant free magnet, a customized split pair coil manufactured by former ACCEL Instruments (now BRUKER ASC) offers a 100 mm room temperature bore, with a coil separation of 30 mm giving an aperture angle of  $\pm$  5° and 330° horizontal access. The magnet provides vertical fields up to 7,5 T at the push of a button. The field homogeneity is better than 0.1 T. The compact dimensions and the large room temperature bore allow for applications with differing modular set ups.

The CCR and the dilution insert have been developed in house by the FRM II sample environment group. The top loading cryostat (CCR) offers a sample tube of Ø 50 mm. A customized pulse tube cold head (PTR), manufactured by former VeriCold GmbH (now Oxford Instruments) provides sample temperatures ranging from 3.2 K up to 300 K. Cool down from RT to 3.2 K takes about 4.5 hours. Since early 2010, a new design has been developed at FRM II, using PTR's manufactured by Sumitomo Heavy Industries, Cryogenics Division, showing a considerably improved performance. Aside from standard sample sticks, rotary sample sticks or gas adsorption sample sticks and high temperature sample sticks are available, the latter allowing temperatures of up to 700 K. To reach for very low temperatures <sup>3</sup>He/<sup>4</sup>He dilution inserts and <sup>3</sup>He-inserts offer base temperatures down to 50 mK and 400 mK respectively (without beam load). The inserts are identical in construction down to the Joule Thomson stage needed to liquefy the incoming and precooled <sup>3</sup>He gas or mixture respectively. Only the low temperature stages are different, the <sup>3</sup>He stage is designed at FRM II the <sup>3</sup>He/<sup>4</sup>He dilution stage is manufactured by ICE Oxford.



The complete liquid-refrigerant free setup at PUMA: the 7.5 T vertical field magnet, top-loading crystat and  $^{3}$ He/ $^{4}$ He dilution insert.

Being in routine operation now, the system complements the other low temperature equipment and may be requested by users in their upcoming proposals.

We thank the experimental team for their assistance and patience, making this first user experiment with this system a success.

Jürgen Peters FRM II



## **RESEDA – Two arms and more**



Fig. 1: Overview of RESEDA during final improvements at the second spectrometer arm.

After the commissioning phase of the instrument in 2008, the Neutron Resonance Spin Echo (NRSE) spectrometer RESEDA started its routine operation about two years ago. Recently, an upgrade of RESEDA was finished, making use of a special feature of the NRSE method namely enabling experiments with two secondary spectrometer arms.

The Neutron Spin Echo (NSE) method relies on neutron spin precession in magnetic field regions. The spin precession angles before and after the sample are compared. If inelastic scattering happens, the precession angles are not equal, and the final spin polarization is decreased. Measurement of the final polarization provides information about the presence and strength of inelastic processes at the sample. Similarly as at standard NSE spectrometers RESEDA was first operated with one primary and one secondary spectrometer arm, where spin precession takes place. However, in contrast to NSE, NRSE allows the straightforward installation of additional secondary spectrometer arms. The magnetic stray fields of the individual arms can be reduced to a nonsignificant level by means of µ-metal shielding, thus not disturbing the proper operation of the neighbouring spectrometer arms. This feature of NRSE has been used at RESEDA by doubling its secondary spectrometer, including all parts inside the µ-metal shielding and the analyzer and detection system. For this purpose, two renewed NRSE coils and regular spin echo coils used for measurements at small spin echo times have been installed and put into operation thus allowing NRSE operation with a doubled solid angle for neutron detection.

RESEDA is typically used in either of two different configurations:

- For studying dynamics at small scattering angles (NRSE-SANS) only one spectrometer arm is used due to geometrical restrictions. In return 3-d polarization analysis is available. Moreover, the background can be reduced by tight beam collimation. For demanding experiments at small scattering angles or low scattered intensity, the NRSE coils can be removed, reducing the amount of material in the beam and further minimizing the background.
- For the study of dynamical properties in samples of soft matter and in glasses, the measurements are



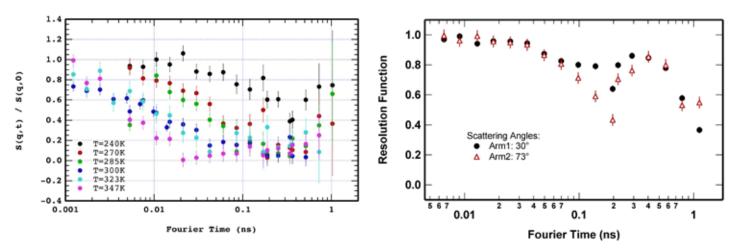


Fig. 2: Intermediate scattering function S(Q,t) measured on a system of water confined in hectorite clay (left, plot from experimental report of experimental report 2302). Resolution function of RESEDA at 5.5 Å using both secondary spectrometer arms (right).

conducted at larger momentum transfer. Hence both spectrometer arms are being used.

Within the two arm configuration, scattering angles up to 100° can be used to study the dynamics on small length-scales. Typical experiments were performed in 2009 on the temperature dependent dynamics of a polymer melt and the dynamics of water in porous media. An example of data acquired at different temperatures is shown in figure 2 (left). According to the thermal energy, the dynamics are taking place at different decay times. For these experiments, the instrument resolution was optimized simultaneously in both secondary spectrometer arms. The typical shape of the resolution function at the neutron wavelength 5.5 Å is also shown in figure 2 (right). The dip at about 150 ps is due to the intersection between NSE and

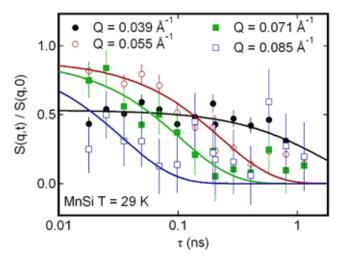


Fig. 3: Typical spin echo data from MnSi as measured at RESEDA. The data are corrected for the instrumental resolution. The measurements have been performed at 29 K.

NRSE. By means of NSE, the small spin echo time region is covered. To decrease the spin echo times further, the neutron velocity selector can be tilted, in order to reach wavelengths as small as 3.5 Å.

In the NRSE-SANS setup, RESEDA is typically used for the investigation of magnetic phase transitions, where the dynamics evolves strongly with temperature. The typical time scale of the magnetic fluctuations matches the spin-echo times available at RESEDA well. Figure 3 shows as a typical example the use of the NRSE-SANS setup for studying the dynamics of the paramagnetic fluctuations in manganese silicide (MnSi). MnSi is a 3d intermetallic compound exhibiting single-handed helical magnet order with a period of approximately 180 Å below the ordering temperature  $T_c = 28.85$  K. Above  $T_{c}$ , long range magnetic order is lost. However, chiral fluctuations still persist. The high flux of RESEDA near a wavelength of 5.5 Å allows a clear identification of the magnetic fluctuations. Around the ordering vector at  $Q = 0.039 \text{ Å}^{-1}$ , the fluctuations are very slow, they decay within about 1.5 ns. With increasing Q, the fluctuations decay very quickly.

Further NRSE experiments proposed by scientists from Germany, England and Switzerland have taken place in the meantime. The requirements and the possible setups of the instrument are discussed between the user and the instrument scientist before the experiment, and the optimum setup is decided together. The variability of RESEDA will be further improved in 2010 by the installation of the MIEZE technique, a NRSE option where the intensity of the neutron beam is modulated using NRSE coils running at different frequencies.

> Wolfgang Häussler FRM II



### **BMBF** funded projects at the FRM II

#### New period from 1.7.2010 to 30.6.2013 has started



Fig. 1 The new polarized cold three-axis instrument KOMPASS.

Verbundforschung describes the financing mode of basic science endeavours by the German federal ministry for education and reserach (BMBF) where the topics are specific to large-scale research, of supraregional, international or fundamental significance and where large-scale equipment for research are required. The section dealing with condensed matter research is subdivided in the types of radiation used, namely synchrotron and free electron lasers, neutrons and charged particle beams.

In the next three year period, starting from July 1<sup>st</sup>, 2010, several projects will be funded to build new instruments and equipment at the FRM II. The total sum of the funding is about 10 Million Euro.

The projects to build new instruments are a follow-up project for the cold threeaxis instrument (fig. 1) KOMPASS (Markus Braden, Universität zu Köln and Peter Böni, Technische Universität München) and the time-of-flight powder diffractometer PowTex (Richard Dronskowski, RWTH Aachen and Bent Hansen, Universität Göttingen). The very successful setup to use polarized neutrons on the single crystal diffractometer HEIDI will become an independent instrument using the beam port SR9a on the hot source (Georg Roth, RWTH Aachen).

A complete new instrument will use the transmitted beam of the PowTex instrument to investigate samples at extreme conditions, i.e. high pressure and high temperatures. This instrument will be built by the Geozentrum Bayreuth in the institute of Hans Keppler. Figure 2 shows a similar high pres-

sure device, recently installed in Bayreuth. Using time-of-flight neutrons, a complete diffraction pattern even under the restricted space condition will be possible. In addition the transmitted beam through the sample will be used for in-situ radiography which is suited to investigate for example, viscosities at these extreme conditions.

The three-axis instrument Puma will be equiped with a sophisticated polarization analysis. Funding for this project was attracted by the Universität Göttingen in the institute of Götz Eckold. Instrumental development on the time-of-flight reflectometer REFSANS will introduce complemen-

tary methods using a dual polarisation interferometer (Joachim Rädler, LMU München). Last but not least the development of new high power magnets will be initiated by a project of the Technische Universität München, led by Christian Pfleiderer.

In the subdivision of particle beams the consortium POSIMETHOD has been successful to acquire funds to further develop the use of positrons at the high intensity source NEPOMUC at the FRM II. The collaboration consists of institutes from the Universität der Bundeswehr (Günther Dollinger, Walter Hansch), Technische Universität München (Peter Böni), Universität Halle (Reinhard Krause-Rehberg) and Universität Kiel (Klaus Rätzke).

Jürgen Neuhaus FRM II



Fig. 2: Multi-anvil press at the Geozentrum Bayreuth.



### Novel electrostatic levitation furnace

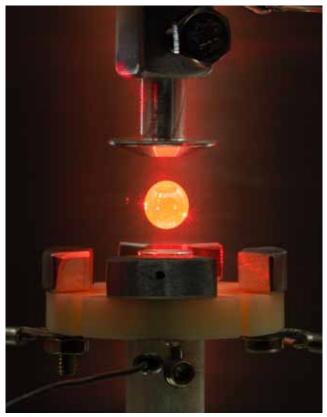


Fig. 1: Electrostatically levitated freely suspended liquid Ti<sub>39.5</sub>Zr<sub>39.5</sub>Ni<sub>21</sub> dropled with a mass of 0.6 g at a temperature of 1150 K.

If performed in crucibles, neutron scattering experiments on liquids have so far been limited to low melting and/or chemically fairly inert systems and to temperatures above the melting temperature. In order to give access to the study of refractory and chemically reactive melts, containerless processing techniques are required. The absence of a sample holder makes it not only possible to extend the accessible temperature range to high temperatures of up to 2500 K, but also into the metastable regime of the undercooled liquid several hundreds of Kelvin below the equilibrium melting point, due to the avoidance of heterogeneous nucleation at crucible walls. In scattering experiments the avoidance of any crucible materials has the additional advange of an impoved signal to background ratio. The first studies of atomic dynamics, of metallic melts prepared using a containerless process, by quasielastic neutron scattering have been performed three years ago by utilization of the electromagnetic levitation technique at the time-of-flight specometer TOFTOF of FRM II. This containerless processing technique, however, is restricted to electrically conductive materials. Moreover, levitation and heating of the samples are coupled, which limits the minimum accessible sample temperatures and requires a process of the samples under a cooling gas atmosphere. The latter bears the risk of contamination of the samples by impurities from the gas atmosphere, which may reduce the undercoolability of the samples by heterogeneous nucleation.

As a joint effort of the Institut für Materialphysik im Weltraum at DLR in Köln and the FRM II a novel sample environment based on the electrostatic levitation technique has been developed that overcomes these limitations. Samples of up to 6 mm in diameter are levitated under ultra-high vacuum conditions using electrostatic forces that are adjusted utilizing an active sample position control. By laser heating temperatures in the range between ambient temperature and more than 2500 K are accessible. The first quasielastic neutron scattering experiments using the new sample environment have been successfully performed on TOFTOF in May 2010. Figure 1 shows a picture of a freely suspended liquid  $Ti_{_{39.5}}Zr_{_{39.5}}Ni_{_{21}}$  dropled with a mass of 0.6 g, processed in the electrostatic levitator at a temperature of T = 1150 K.

The experiments performed during the beamtime allowed the study of atomic dynamics in different Zrbased alloy melts as a function of the temperature extending into the metastable regime of an undercooled melt. As an example, figure 2 shows a raw time-of flight spectrum measured for a  $Zr_{64}Ni_{36}$  melt at T = 1478 K that was acquired within a range of scattering angles of  $59^{\circ} < 2\Theta < 74^{\circ}$  using a wavelength of 7 Å. Also shown is the spectrum of the empty sample environment. Due to the containerless processing setup, the background is about 2 orders of magnitude smaller than the signal measured with sample.

Dirk Holland-Moritz DLR Köln

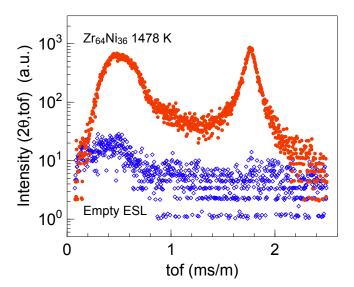


Fig. 2: Raw time-of flight spectrum measured for a  $Zr_{64}Ni_{36}$  melt at T = 1478 K that was acquired within a range of scattering angles of  $59^{\circ} < 20 < 74^{\circ}$ . Also shown is a spectrum of the empty levitator.

### **Events**



# Nondestructive testing of industrial components

#### From ultrasound to neutrons: 3<sup>rd</sup> VDI expert forum in Garching

Presenting various examples of recent industry related projects, the 3<sup>rd</sup> expert forum organised by the advisory board for Application Oriented Nondestructive Materials and Component Testing of the VDI gathered more than 60 participants at the faculty of mechanical engineering in Garching on April 13<sup>th</sup>, 2010. During the one day meeting, chaired by Ralph Gilles, 8 lectures were given on a wide range of topics in order to discuss the pros and cons of different methods for nondestructive investigations.

A well prepared door of a car has been investigated with different imaging methods. Inga Wehmeyer from Ford explained the requirements of tight sealing in the fabrication process in order to prevent corrosion on inner surfaces of the doors. Different methods such as ultra sound, thermography and x-ray and neutron radiography were applied to detect intentionally introduced defects including things like inhomogeneous distribution of the glue. With the high contrast of the neutrons caused by the hydrogen containing glue, a detailed analysis could be undertaken to serve as a reference for the other methods. In this case ultrasound and thermography could not provide the required sensitivity to the inner structures, x-rays might be feasible for the industrial inspection in a factory.

Overviews of ultrasonic and thermography methods were given by Ulrich Bücher (Olympus) and Gerhard Busse (IKT Stuttgart) respectively. These widely used inspection techniques deliver remarkable results under well defined conditions where the interpretation of the obtained signal can be assigned unambiguously to known defects. Especially the depth resolving lock-in thermography exited thermally or by ultra sound revealed impressive pictures. The continously ongoing development of the ultra sound testing on the other



Fig.2 Visiting the stress and texture diffractometer STRESSSPEC.



Fig.1 Stairways to heaven of nondestructive testing.

hand can cover a wide range of materials from carbon fibre reinforced plastik (CFRP) to steels. Using phased array techniques quantitative measures are possible for hidden defects for example in CFRP even on large objects like parts of an aircraft or wings of wind turbines. The examples presented for both methods showed impressively how imaging of structures in nondestructive testing is used in a production environment even for large test objects.

Using the neutron radiography station ANTARES at the FRM II the Institut for thermo fluid dynamics of the TU Hamburg-Harburg investigated the moisture take up of an airplane wall. Andreas Joost reported on the measurements simulating the different conditions of a flight, from high temperatures on the ground to -50 C

### **Events**





Fig. 3 Lively discussions during the break on the possibilities of imaging using fast neutrons.

during the flight phase at high altitudes. A quantitative analysis of the weight increase due to water take-up could be undertaken. Future modelling of the water take-up will provide a detailed analysis of weight changes on the entire aircraft.

Combining different types of steel by welding is a common problem in plant engineering. In order to investigate thick wall tubes, Mathias Büttner from the

question how to validate and interpret the obtained results. Bernd Valeske from the IZFP in Saarbrücken presented a broad overview of methods used for the quality control in industrie to ensure safe and reliable operation of technical constructions.

Last but not least, Bodo Gudehus from the WIS in Munster presented the testing of radiation damage on MOSelectronics induced by fast and thermal neutrons. For the testing the group has used the converter facility as well as the radiography station ANTARES at the FRM II. In situ monitoring of different kinds of defect showed remarkable differences in the number of errors depending on the energy of the neutrons.

A final panel discussion, chaired by Heinz Voggenreiter, summarized the different aspects of nondestructive testing presented in the talks and led to a lively discussion with the audience. The forum ended with a visit of the experimental facilities of the FRM II.

> Jürgen Neuhaus FRM II

MPA, Stuttgart used the high penetration depth of neutrons at the instrument STRESSSPEC. With a sample volume of 5 x 5 x 5 mm<sup>3</sup>, inner stress could be investigated in the 3 cm thick wall of the tubes near the welding area. Using finite element simulations the observed tensions could be modeled sufficiently precise in axial and circumferential directions of the tube.

Pushing further the limits of sophisticated nondestructive materials testing always raises the



Fig. 4 Summarizing and networking during the panel discussion.



## **SNI 2010**

#### February 24th-26th, 2010 - Berlin

More than 700 hundred scientists joined the German conference on Research with Synchrotron Radiation, Neutrons and Ion beams on Large Scale Facilities (SNI 2010) on February 24<sup>th</sup>-26<sup>th</sup> in Berlin. The conference, organized every four years, presents a current overview of research using the three different probes and informs delegates about the latest developments and projects at the German facilities. In three parallel sessions including oral presentations and two extended poster sessions an impressive overview of research with synchrotrons, neutrons and ion beams was given. The presentations in all areas of science demonstrated the extensive use of each probe and the request by scientists for their availability.

A large number of oral and poster presentations showed the activities of the users at FRM II and JCNS ranging from magnetism to biophysics, material research and soft matter research. Within the presentations of future activities in synchrotron, neutron and ion beam research the discussion on the future European Spallation Source ESS in Lund, Sweden, attracted a large number of participants and information presented on the latest developments on X-FEL facilities world wide within the plenary talks were highly welcome.

After three days of intense discussions at this very successful meeting the community is looking forward to the next SNI in 2014.

Thomas Gutberlet JCNS





## **JCNS School on data analysis for QENS**

#### April 28th-29th, 2010 - Garching



Despite warm sunshine on April 28<sup>th</sup> and 29<sup>th</sup> about 20 participants from various universities and research centres in Europe attended the recent first JCNS school on data analysis organised by Joachim Wuttke, instrument scientist at the backscattering spectrometer SPHERES at JCNS at FRM II. The school was dedicated to users and instrument scientists of neutron time-of-flight

and backscattering spectrometers, who like to use or do use the program FRIDA in their work. The highly motivated participants were introduced into the basic concepts of quasi-elastic and inelastic neutron scattering and worked on data reduction and analysis with the program package SLAW and FRIDA. Interactive visualisation and conditioning of pluri-dimensional data were discussed, the foundations and practice of curve fitting was done and typical problems in analysing quasielastic neutron data were shown.

In discussion with the participants problems and improvements of the programs were talked about. In the evening all partcipants were invited for dinner in one of the famous Munich beer gardens to relax. Everyone enjoyed the fruitful and encouraging atmosphere of this school, which was organized for the first time at JCNS.

Thomas Gutberlet JCNS

### **Events**

### **Events**



### The FRM II at night

#### Lange Nacht der Wissenschaften: May 15th, 2010 - Garching



One group was traditionally reserved for children of staff members. They got an interesting insight into the operation of the reactor while visiting the FRM II crew in the control room.

428 visitors came to see the neutron source on the "Lange Nacht der Wissenschaften". The tours on Saturday May 15<sup>th</sup> from 16 to 23 h were fully booked after 1.5 hours. At peak times, people lined up throughout the foyer of the Physics Department of the Technische Universität München through the entrance doors.

While waiting, the visitors could watch a film by the company itg, showing the production of radioisotopes at the FRM II. Franz Michael Wagner explained the safety features at the neutron source and the natural sources of radiation at the booth of the FRM II radiation protection.

The four lectures given by FRM II scientists, Winfried Petry, Birgit Loeper, Christoph Hugenschmidt and Ralph Gilles, were well attended. Among the audience and the visitors were several guests from the ecumenical church day in Munich.

Andrea Voit FRM II

# **ERF and GENNESYS meetings in Barcelona**

#### FRM II joins the group of European Research Facilities and Gennesys congress

Large scale research infrastructures work together on an European level in two different organisations. Intergovernmental scientific research organisations cooperate in the EIROforum:

#### www.eiroforum.org

while national facilities are organised in the ERF (European association of national Research Facilities laboratories):

#### www.europeanresearchfacilities.eu

During the last meeting on May 26<sup>th</sup> 2010 in Barcelona, the FRM II joined this organisation as an associated member. Regular seminars are organised by the ERF, the next one entitled "Human Capital for Modern RIs" which will take place at the PSI on 19<sup>th</sup> of October 2010.

Just after this meeting the GENNESYS, International Congress on Nanotechnology and Research Infrastructures:

#### www.gennesys2010.eu

took place from 26<sup>th</sup> to 28<sup>th</sup> May in the CCIB congress center in Barcelona. About 250 participants attended to discuss the future development of nanotechnology in Europe pointing out the grand challenges to be addressed by research using synchrotron and neutron facilities. The sessions covered general aspects of the research infrastructure in Europe as well as nano-material design, industrial innovations and societal challenges. The congress concluded with summarising the European and international needs for developing nanotechnology including research and educational concerns. During the breaks fruitful discussions took place at the presentation of the FRM II and the JCNS booth.

> Jürgen Neuhaus FRM II



Discussing new experiments using neutrons made in Garching.



### Materials science with positrons

#### Looking for defects with CDBS

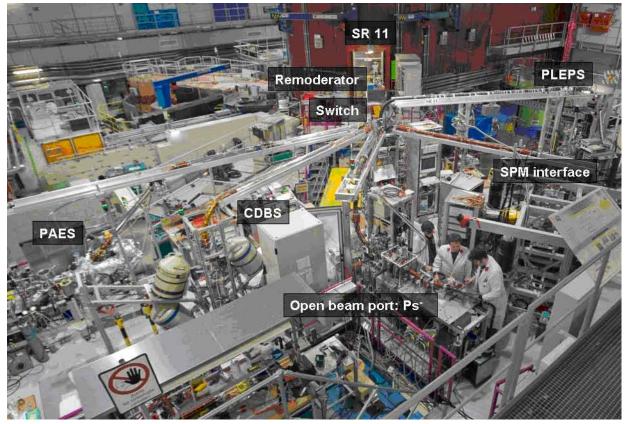


Fig. 1: The positron beamline and the instruments at NEPOMUC. The present experiments are performed at the coincident Doppler-broadening spectrometer – CDBS.

The neutron induced positron source NEPOMUC at the FRM II provides the most intense low-energy positron beam in the world of almost 10<sup>9</sup> moderated positrons per second. Presently, the positron beam can be switched to five different experimental stations shown in figure 1.

In solid state physics and materials science the positron – the anti-particle of the electron – is applied as a high sensitive nano-probe for defect spectroscopy. Its high sensitivity to open volume defects is correlated to a local attractive potential which traps the positron until it annihilates together with an electron. Since the annihilation radiation contains information of the electron momentum distribution at the positron annihilation site, the presence of defects on an atomic scale is experimentally accessible.

As one example, the coincident Doppler-broadening spectrometer (CDBS) allows the examination of defects near the surface and in the bulk up to a few  $\mu$ m according to the maximum positron implantation energy up to 30 keV. With a positioning device 2D-scans of the sample under investigation can be performed with a lateral resolution of 300  $\mu$ m. These spatial re-

solved measurements are particularly suited to image lattice defects such as vacancies or vacancy clusters. Moreover, element specific information in the area surrounding a defect is gained which plays a major role e.g. for the understanding of precipitation growth in metallic alloys. Spatial resolved positron studies using a step width of 1 mm were performed recently on plastically deformed Al and on friction-stir welded (FSW) Al alloys.

Plastically deformed regions of an Al sample were investigated after a tensile test which leads to the formation of lattice defects on an atomic scale. The spatially resolved measurement clearly reveals the presence of such defects in a central region according to the color legend of figure 2. In particular, the defects could be detected even in regions, where no deformation was seen with an optical microscope. Moreover, the obtained S-parameter could be correlated to the absolute value of the mechanical stress which was acting locally when the mechanical load was applied, and which was hence responsible for the defect creation.

In a second study, we investigated the defect distribution near a welding joint in AlSi<sub>a</sub>Cu<sub>a</sub>. This used a



friction-stir welding process which produces the required heat input for joining components; the friction occurring between a rotating tool and the work piece. Besides the possibility to weld different materials like Al and steel, this welding technique has the advantage that pore formation or hot cracking can be avoided, as no high temperature gradients or a liquid phase with the subsequent solidification process occur.

As shown in the positron scan of figure 3, FSW leads to the production of defects in the thermo-mechanical affected zone. Confirmed by line scans with improved lateral resolution of 250  $\mu$ m (not shown), the spatially resolved defect distribution revealed that the material in the joint zone becomes completely annealed during the welding whereas at the tip, annealing is secondary while the deterioration of the material due to the tool movement prevails. This might be responsible for the increased probability of cracking in the heat affected zone of friction stir welds.

#### Acknowledgments

The author thanks P. Gebhard of IWB of the Technische Universität München for the preparation of the FSW samples.

Christoph Hugenschmidt TUM

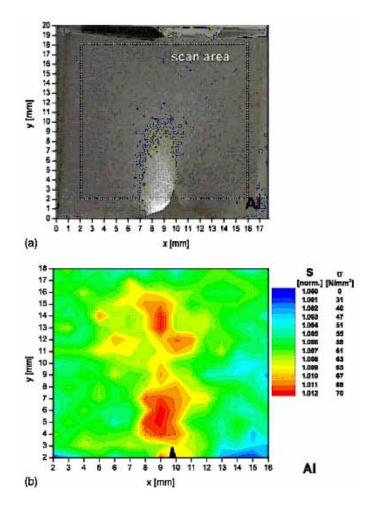


Fig. 2: Asymmetrically deformed AI sample with a notch after tensile test: optical image (a), and 2D S-parameter scan with a positron energy of 26 keV with the derived stress values (b).

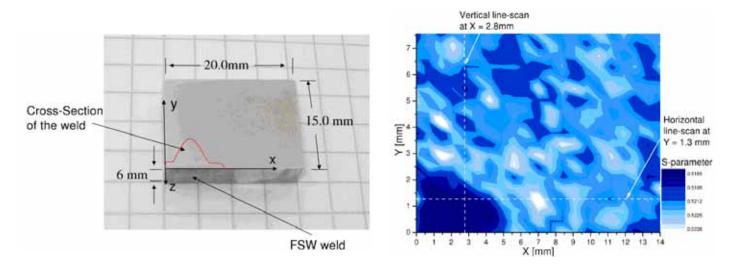


Fig. 3: FSW sample of AlSi Cu,: The red dotted line indicates the shape of the cross-section of the FSW weld where the pin was moving along the z-direction (left). The 2D S-parameter scan with a positron energy of 30 keV (right). The dark area clearly corresponds to the FSW induced defects in the sample.

#### www.frm2.tum.de/nepomuc



# What's the matter with matter?



Fig. 1: The setup of the xenon experiment in the neutron guide hall east of the FRM II.

The "early" physics of the universe – the understanding of the origin and the nature of matter – is the focus of interest of the junior research group "Fundamental Physics with Neutrons", led by Peter Fierlinger.

In our everyday experience matter is something that is just there: matter forms our planet, the living and the inanimate nature on earth, our solar system, all stars and galaxies in the cosmos. However, we cannot take the presence of matter for granted. "How matter came into existence is one of the major unsolved mysteries in the history of the universe", explains Peter Fierlinger. There is evidence that shortly after the big bang there was an anti- material counterpart for each particle, e.g. the positron for the electron or the anti-neutron for the neutron. According to the laws of physics, particles and anti-particles cannot easily co-exist, as they destroy or annihilate each other when they meet, thereby releasing energy.

In this scenario neither matter nor anti-matter would have survived and our universe today would consist of pure energy. "Our existence is the proof that somehow the symmetry of matter and anti-matter must have been disturbed", says Fierlinger. "At some point, there was a surplus of matter versus anti-matter that escaped annihilation." According to current knowledge there was one extra-matter particle in a number of one billion particles - today's matter-dominated universe is the result of this tiny numerical difference. Looking at the origin of matter one might consider it an accident that just happened. This of course is an unsatisfactory explanation. The starting point for Fierlinger's investigations is the research of Paul Dirac, who predicted the existence of mysterious anti-particles. These have the same weight as 'normal' particles and the same amount of charge but with opposite sign. After such particles were indeed found a few years later, Wolfgang Pauli stated that this similar behaviour follows from fundamental symmetric properties of the universe: the reversal of charge (C), the reversal of the spatial coordinates (P) and the reversal of time (T). This relation is called the CPT symmetry. Although scientists assume that our universe perfectly conserves these symmetries, some combinations of theses symmetries must have been violated to create the predominance of matter.

Peter Fierlinger explains how scientists deal with this riddle: "Today's research in this field focuses on finding examples of symmetry breaking in fundamental phys-



use proven methods,

we develop a com-

pletely novel measuring technique for

xenon". According to

Peter Fierlinger, this

approach opens new

possibilities for inves-

effects in EDM exper-

iments. When asked,

what would happen

to the neutron EDM

experiment if there

were promising re-

sults from xenon, he

says: "A measured

EDM would be a

tion of something

spectacular

systematic

indica-

tigating

ical systems." One different route to find more broken symmetries is to look for electric dipole moments of fundamental systems: Due to mathematical calculations we know that a permanent electrical dipole moment (EDM) in a particle would hurt both the P and the T component in the CPT equilibrium. "Therefore, time, the first experiments will be performed at the PSI neutron source.

Fierlinger's group doesn't exclusively concentrate on neutrons. Another suitable experimental system is based on an isoptope of the inert gas xenon (129-Xe). "In contrast to the neutron EDM experiments that will

we search for an EDM in two different experimental settings", adds Fierlinger.

A very good candidate for EDM search is the neutron due to its relatively simple composition. Therefore, Fierlinger and his are group currently setting uр experiments at the Excellence Cluster Universe and at the Paul Scherrer Institut (PSI) in Switzerland. Peter

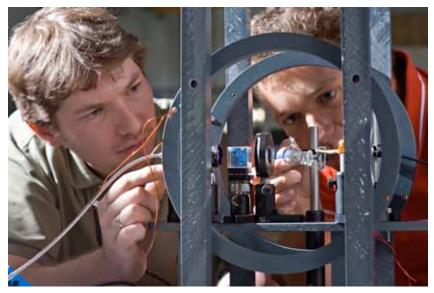


Fig. 2: Peter Fierlinger (left) and PhD student Florian Kuchler (right) optimize the setup.

Fierlinger: "The neutron EDM experiment is based on trapped ultra-cold neutrons, these neutrons are cooled down to low energies and move with the velocity of a bicycle rider – allowing for much easier investigations than in 'normal', fast moving neutrons". The FRM II is building a new plant for ultra-cold neutrons that will start operation in 2011. In the mean-



Fig. 3: Interior of the cryostat for the xenon EDM measurement.

unknown, but the observation of this effect in one system alone does not tell us enough about the underlying physics." Therefore it would make the neutron EDM measurement even more interesting. Xenon is also the connecting link to a different research topic Fierlinger is involved in: The hunt for the value of the neutrino mass, and whether the almost massless neutrino is its own antiparticle. "Like electrons, neutrinos are elementary particles known as leptons", says Peter Fierlinger. "There are indications that the neutrino doesn't have an explicit counterpart as is the case with electron and its antiparticle positron." Using the isotope 136-xenon, scientists are aiming at detecting neutrinoless double beta decay, a special case of radioactive decay. This is expected to be very rare, but it should answer the question of mass and nature of the neutrino. The experiment is currently being built in an international collaboration based at Stanford University.

The JRG "Fundamental Physics with Neutrons" is situated in Garching with strong ties to PSI, PTB Berlin, Stanford University and Argonne National Lab.

Only recently, the Cluster Universe has been granted another 3.72 million Euros by the German Research Foundation DFG. Part of the funding will be used for the EDM-experiment and the construction of the ultracold neutron source.

> Barbara Wankerl TUM



# **Ancient Charm**

In 2006, an EU Project called ANCIENT CHARM (Analysis by Neutron resonant Capture Imaging and other Emerging Neutron Techniques: new Cultural Heritage and Archaeological Research Methods) was started in frame of the FP 6 program and was successfully finished in December 2009.

The aim of the AC project was to develop three complementary neutron-based analysis techniques into noninvasive methods for 3D tomographic imaging of the elemental and phase composition of cultural heritage and other complex archaeological objects:

- Neutron Resonant Capture Imaging combined with Neutron Resonance Transmission (NRCI/NRT)
- Prompt Gamma Activation Imaging combined with cold Neutron Tomography (PGAI/NT)
- Neutron Diffraction Tomography (NDT)

Four large neutron facilities have participated at this project:

- The Prompt Gamma Activation Analysis (PGAA) facility at the Budapest research reactor (PGAI/NT).
- The PGAA and NT facility at the FRM II research reactor in Garching, Germany (PGAI/NT).
- The pulsed neutron beam of GELINA, Geel, Belgium (NRCI/NRT).
- The 800 MeV pulsed spallation neutron source at ISIS, Didcot,United Kingdom (NDT).

This report will focus on the PGAI/NT experiments performed at the PGAA instrument at FRM II.

Prompt Gamma-ray Activation Analysis (PGAA) is a powerful tool for the non-destructive, bulk elemental analysis of samples from several fields of research. In the frame of the European Ancient Charm project it

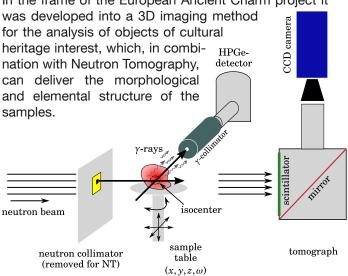


Fig. 1: Principle of the PGAI/NT method.

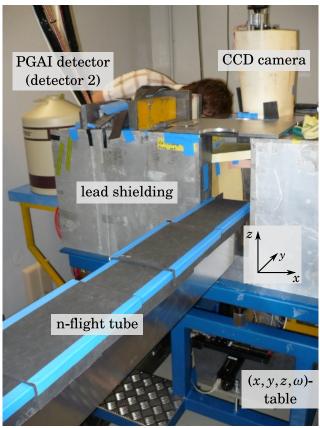


Fig. 2: The PGAI/NT setup at FRM II.

When irradiated by cold neutrons, materials emit characteristic gamma-rays which can be used to identify and quantify the composition of the analysed samples. By collimation of the neutron beam and the gamma-ray detector (fig. 1) the samples, which are placed on a (x,y,z,w)-moving table, can be scanned position by position, resulting in an elemental map of the sample. The spatial resolution that can be obtained with this method is partly dependent on the measured elements but is of the order of a few mm<sup>3</sup>. To reduce the necessary measurement times it is advisable to use a previously performed Neutron Tomography, to limit the sample scanning to some interesting positions inside the sample. For the correct sample positioning and later allocation of the measured positions in the Neutron Tomography reconstruction, universal sample holders, which provide a common reference frame for both measurement methods, are used.

A combined PGA Imaging and Neutron Tomography setup was installed at the FRM II PGAA instrument (fig. 2) and first measurements on real cultural heritage objects were performed here. An ancient disc fibula (fig. 3(a) and (b)) from the second half of the  $6^{th}$  century – a central object of interest of the Ancient





Fig. 3: Disc fibula from 6<sup>th</sup> century. Left: front, right: side.

Charm Project - was brought to FRM II for detailed analysis by scientists from the Hungarian National Museum in Budapest. The main structure of the fibula appeared to be made out of iron, covered by gold lined almandine ( $Fe_2Al_2Si_3O_{12}$ ) inlays on the front. The back-plate of the fibula was supposed to be bronze. Because the iron structure of the fibula and the gold lining of the almandine inlays are quite uncommon for a fibula of this origin and period, this object was of special cultural heritage interest.

After a Neutron Tomography of the fibula was done at the PGAA setup the upper left quadrant was scanned with 3D-PGAI in three layers. Due to the symmetry of the object this quadrant was considered as representative for the other quadrants and the results from the measured layers were extended to the remaining parts of the fibula during the analysis. The positions of these layers were chosen to cover the inlays of the fibula, the inner part of the fibula and the front of the back-plate. The Neutron Tomography proved to be an invaluable tool for the selection of these measurement positions. shown in figures 4(a)-(e). As we can see from the silver distribution (fig. 4(a)) the almandine linings seem to be not solely out of gold, but also do contain some amount of silver. Gold can be seen all-over on the outer layers of the fibula (fig. 4(b)), which is probably due to some residuals of a former gilding layer. The copper distribution (fig. 4(c)) reproduces the position of the back-plate of the fibula. Iron was detected over the full fibula volume (fig. 4(d)). Especially the iron abundances found at the back layer may indicate another iron plate below the bronze back-plate. Sulfur

(fig. 4(e)) and hydrogen were identified as components of the filling material.

The results of this measurement showed some interesting properties of the fibula which can now help to identify the manufacturing techniques and the exact provenance of this object.

The PGAI/NT measurements during the Ancient Charm Project proved the general feasibility of this technique at the high intensity cold neutron flux available at the PGAA instrument and were the motivation for further development and improvement of PGAI/NT method at FRM II with the aim to install it as a permanent (exchangable) part of the present PGAA setup.

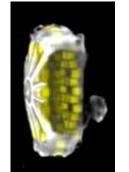
Financial support of the Ancient Charm project by the European Community "New and Emerging Science and Technology" Contract No 15311 is acknowledged.

Ralf Schulze Universität zu Köln

Some exemplary results of these measurements are

(b) Gold



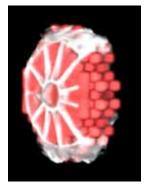


(a) Silver

Fig. 4: Elemental abundances in the disc fibula.



(c) Copper



(d) Iron www.frm2.tum.de/pgaa



(e) Sulfur.



# <sup>3</sup>He polarization analysis for biological samples

A team of JCNS scientists lead by E. Babcock. A. loffe, A. Radulescu, and V. Pipich have been working on implementing new measurements techniques for the JCNS SANS instrument KWS-2. This technique uses polarization analysis to directly measure the coherent scattering pattern from biological samples.

Using SANS to probe the structure of biological samples requires precise determination of the parameters used to model or fit the scattering pattern generated by the sample. These parameters include forward scattering, structure, size, and background. The first three parameters are related to coherent scattering and the fourth arises from incoherent scattering. The coherent

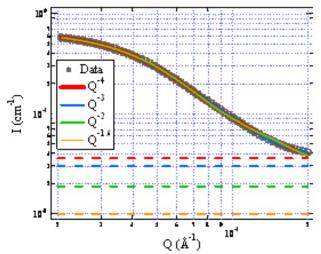


Fig. 1: A simulated scattering curve, open grey circles, with a  $Q^{-4}$  form factor. The four solid lines, which are nearly impossible to distinguish because they overlap, red, blue green, and orange, are Beacauge fits with the form factor held to power laws of -4, -3, -2, and -1.6 respectively. The dotted lines are the corresponding value of the background from the individual fits of the same color. From data like this alone, accurate determination of the form factor would not be possible.

scattering gives information on the samples structure whereas the incoherent scattering is just background competing with the desired information. Normal biological samples contain hydrogen which produces incoherent scattering, thus the sample itself creates an intrinsic background.

Figure 1 shows a cartoon example of how this background leads to ambiguity in the parameters determined from a given protein sample. In the models or fits of such data, the parameter for the background is coupled to the other parameters. Changing the assumed level of background can infer very different form factors, i.e. the samples shape, which comes from the power law of the scattering, which then couples to the determination of other important parameters such as the samples size. Essentially any of the possible ranges for the form factor can be fit to the curve, which was generated for a Q<sup>-4</sup> dependence, i.e. Porod like scattering. The information on form factor is entirely determined in the fit by the data points in the Q > 10<sup>-1</sup> Å<sup>-1</sup> regime for this simulated data. For real data this regime would have low statistical certainty because of the low scattering cross section, and accurate information is hard to obtain. Accurate information in this Q-range is also very crucial for modeling protein form with Monte Carlo simulation.

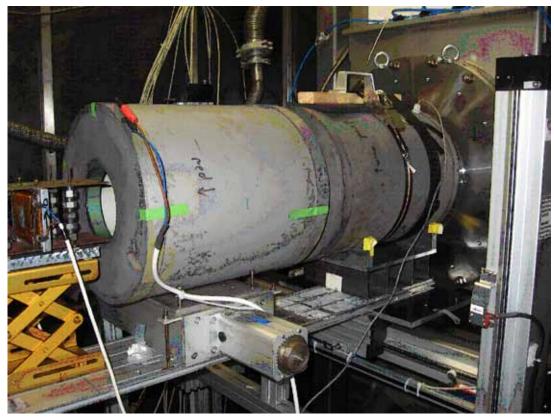
Insufficient knowledge of the background can lead to ambiguity. Since coherent scattering does not change a scattered neutrons polarization, whereas incoherent scattering does, analyzing the scattered neutron spins of a polarized incident neutron beam allows one to distinguish between the coherent and incoherent components, eliminating the ambiguity from background.

Incident beam polarization on KWS2 was available for experiments for several weeks at the end of the 23<sup>rd</sup> reactor cycle at the FRM II. The beam polarization was accomplished with a single super mirror in transmission geometry providing an incident beam polarization of 97 % at 4.5 Å, a Mezei-style spin flipper was used before the sample to flip the incident spin. To provide polarization analysis (PA), a polarized <sup>3</sup>He neutron spin filter was placed after the sample. This <sup>3</sup>He neutron spin filter gave a high polarization efficiency of 95 % at the beginning of the experiments and was 93 % after over 36 hours of measurements.

To achieve this high performance, the <sup>3</sup>He cell and the sample were in the common magnetic environment of a uniform 10 G field provided by a 38 cm long shielded end-compensated solenoid. A second similar shielded solenoid was used for isolation from the external field gradients created by the spin flipper, and for adiabatic rotation of the vertical neutron guide field into the longitudinal direction of the solenoids. This arraignment provided very high performance for the <sup>3</sup>He, giving a total relaxation time of 430 hours for the <sup>3</sup>He polarization during the measurements. A picture of the installation is shown in figure 2.

Polarization of the cell was carried out in the JCNS SEOP laboratory. The initial <sup>3</sup>He polarization of 72 % was the saturated maximum value attainable for the cell used. This level of performance would allow us to operate for 4 to 5 days on a single polarization of the cell with good neutron performance. Since the measurement times for each sample were found to be on the order of an hour or less, many experiments could be performed in this time.





A sample plot of the data obtained is shown in figure 3. Curves for both standard un-polarized measurements, and measurements using PA are shown. The data with polarization analysis clearly demonstrates the removal of the incoherent background and the data can be fit to a model that only contains the coherent scattering components. While this is a straightforward example, for a high-concentration sample (5 % protein in deuterated buffer solution) with Q<sup>-4</sup> form factor, we expect that the technique could become particularly useful in difficult situations. When the Q-range is insufficient to observe the level of the background, the form factor is a lower order power, the sample cannot be measured at high concentrations due to aggregation, or when possible hydrogen exchange between

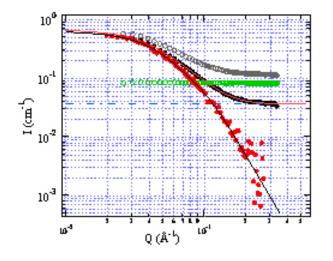


Fig. 2: Picture of the installation on KWS2 showing the two solenoids and the spin flipper.

The sample is inside the solenoid on the right a few cm before the <sup>3</sup>He spin filter cell. Overlap from the vertical field of the spin flipper and the longitudinal field of the first (left) solenoid allow adiabatic rotation of the neutron spin.

the sample and solvent makes correct solvent subtraction and background determinations problematic, PA could provide the needed strait forward solution. Additionally, PA may also enable measurements with only one sample concentration. Other types of samples may require more complex models to de-

scribe the data where removal of the background will similarly remove ambiguity by allowing modeling containing only the coherent scattering.

We would like to thank T.R. Gentile and W.C. Chen for the loan of the cell (called Bullwinkle) used in the measurement. Future plans will focus on developing an optimal magnetic cavity for the J1 cell, made in Forschungszentrum Jülich, to allow in-situ polarization and increase the available Q-range to 0.5 - 0.6 Å<sup>-1</sup> while using standard sample environment. Further, installing a new super mirror polarizer will provide about a 100 x higher flux on the sample, increasing statistical accuracy and lowering measurement time.

> Earl Babcock JCNS

Fig. 3: Sample of the data obtained. The grey/green markers are the data obtained from standard measurements for the sample+solvent and a separate measurement of the solvent respectively. After data treatment one obtains the black/red markers which are the standard SANS signal, and the signal with PA respectively. The red/black lines are fits to the data, and the blue dotted line is the presumed back-ground level from the fit of the standard data. Fitting the PA data to a Beaucage fit with no background, one can obtain the structure dependent fit parameters with no additional assumptions, and the values obtained are consistent with those obtained from standard measurements using several concentrations and no PA. Thus knowledge of the protein is obtained unambiguously with the one measurement employing PA. The data shown is from a sample prepared and by C. Sill who assisted the measurements.



## **Teaching neutron science**

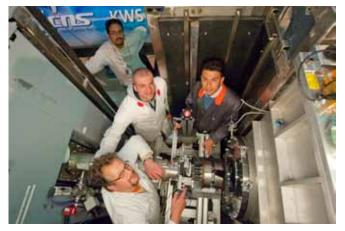


Fig. 1: Latif Sarac (right) did his course work in chemistry at the JCNSinstrument KWS-1 with (from left to right): Henrich Frielinghaus, Frederik Lipfert and Marie-Sousai Appavou.

Teaching neutron science starts early in Garching: From junior high schoolers up to students, everybody can learn anything about neutrons and the related sciences with hands-on experiments, lectures, practical courses, internships, theses or video conferences.

How does a neutron scientist work? Why does he like his job? And what are the neutrons used for? Questions like these are answered in regular video conferences between high schools at the Deutsche Museum and the FRM II. For the first time, Burkhard Schillinger explained

istry at the Graf-Rasso-Gymnasium Fürstenfeldbruck. The 19-year-old applied via Email for the experiment and he was lucky: Henrich Frielinghaus took over the supervision. Latif Sarac investigated an oil-watersurfactant fluid at different temperatures with neutrons under the guidance of diploma student Frederik Lipfert. During his Easter and Pentecost holidays, the grade 12 high school student Sarac spent his time in the neutron guide hall measuring the liguid and writing up his coursework. Meanwhile, Latif Sarac thinks about studying physics instead of chemistry. And Henrich Frielinghaus is happy about the youngest member in his research group: "I am thinking about having more coursework students."

The FRM II also offers several lecture courses to students at the TUM. Tobias Unruh, instrument responsible of the time of flight spectrometer TOFTOF, presents "Neutron scattering and complementary methods I and II" with exercises to graduate students. The nuclear part of the neutron source is covered in a course given by Christoph Morkel of the FRM II entitled "Reactor physics and new concepts in nuclear power engineering I and II". The basics of nuclear physics, the calculations and concepts of reactors are explained to students from mechanical engineering and physics. Finally "Particle physics with neutrons" is held by Peter Fierlinger, who leads the pre-experi-

the tomography and radiography instrument ANTARES to 9th graders (see FRM II News N° 3). Prof. Peter Böni, who will perform his second video conference for school classes, is enthusiastic: "I am surprised about the quality of questions, the kids are asking." He has previously performed a live experiment at the spectrometer MIRA of the Technische Universität München (TUM). The pupils can ask questions during the experiment and afterwards - and



Fig. 2: Peter Böni at the live video conference, seen pupils at the Deutsche Museum.

many made use of this communication method. The aim of the video conference to the neutron source is to fascinate young people about science by giving them an insight into the work of scientists.

Latif Sarac got an even deeper insight, when he performed experiments at the small angle scattering instrument KWS-1 of the JCNS for his coursework in chemneutron source Heinz Maier-Leibnitz is offered to undergraduate students twice a year and is one of the most popular courses with physics students in the 5<sup>th</sup> and 6<sup>th</sup> semester at the Technische Universität München. Many of the students, who get to do hands-on experiments and background theory during two weeks, work as student trainees at the FRM II afterwards or do their master or diploma theses at a

mental group for the ultracold neutron source at the FRM II.

An obligatory part of the lectures is a guided tour of the FRM II to get an impression of the size of the neutron scattering instruments and the reactor.

This is usually, when the students ask about the possibility of practical courses. And there are three each year, lasting several days. The "F-Praktikum" at the



neutron scattering instrument. "So both sides have their profits: We get good students as junior researchers and the students get excellent teaching at real experiments", says Robert Georgii, who organizes the F-Praktikum at the FRM II. The JCNS also offers a neutron scattering course with the annual JCNS lab course open to students from different backgrounds like physics, chemistry or geology. It is part of the curriculum of the Westfälische Wilhelms-Universität Münster and the RWTH Aachen and gives a realistic insight into the experimental technique neutron scattering and its scientific power.

The possibilities after the practical courses are even larger. Graduate students can perform an internship at the FRM II. Several dozen internships are offered every year as well as diploma, bachelor and master theses. An actual overview is published online at www.frm2.tum.de/en/aktuelles/jobs.

> Andrea Voit FRM II



Fig. 3: A camera shows Peter Böni's experiments on MIRA live to the viewers in the Deutsche Museum.

## The FRM II and the ESS sign a collaboration agreement

In a memorandum of understanding, the directors of the neutron source Heinz Maier-Leibnitz (FRM II) in Garching, Germany, and of the future European Spallation Source ESS in Lund, Sweden, defined a farreaching cooperation. The collaboration of the neutron sources will acompass the support during the design and construction phase of the ESS, the strengthening of the European user community as well as close collaboration in research and education. Winfried Petry, Scientific Director of the FRM II, and Colin Carlile, Director of the ESS, signed the document witnessed by the science ministers of



From left to right: Colin Carlile, Charlotte Sahl-Madsen, Wolfgang Heubisch, and Winfried Petry.

Bavaria (Germany) and Denmark, Wolfgang Heubisch and Charlotte Sahl-Madsen, in Copenhagen.

The cooperation envisions that scientists of the Technische Universität München support the ESS during the design phase by means of neutronics calof scientific instruments, sample environment and the development of detectors. Furthermore, both facilities are ambitious to use neutrons for industrial and medical applications in addition to basic science. The ESS will benefit from the FRM II's experiences with industrial and medical projects. Finally, the memorandum of understanding comprises the exchanges of scientists from Bavaria and Sweden for the purpose of education: common neutron workshops and summer schools are intended. Winfried Petry is looking forward to the cooperation with the neutron source in Sweden: "We have an enor-

culations, the construction

mous tradition in designing new instruments for neutron science at the Technische Universität München. Our scientists are eager to support the ESS with their knowledge."

Jürgen Neuhaus FRM II



# Looking at biomembranes with neutron reflectometry



Fig. 1: Sample position of the RESFANS instrument with sample cell in place. Evacuated detector tube and primary beam tube are positioned close to the sample to suppress air scattering.

Lipid membranes are the structural backbone of cellular walls. The main structural motive of this wall is the lipid bilayer of only 4 nm thickness. This bilayer, which is stabilized by hydrophilic/hydrophobic forces, acts as a barrier for ions and larger molecules. Since about one third of all proteins in the cell are associated with the lipid bilayer, the bilayer also acts as a giant switchboard which allows membrane proteins to cluster and transmit signals across it. Sometimes, membrane associated proteins undergo structural changes which have a drastic influence on their interaction and solubility, include pathologic behavior such as massive aggregation as observed for Parkinson and Alzheimer disease.

We aim to study aspects of such lipid-protein interaction by neutron reflectometry and GiSANS. For this purpose, we prepare fluid lipid bilayers on solid supports such as silicon oxide. Here, we benefit from the op-

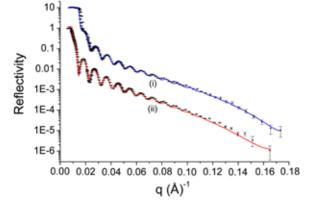


Fig. 2: Reflectivity data using contrast variation. (i) Solid supported lipid bilayer (SLB) in a D<sub>2</sub>O based PBS-buffer solution. (ii) SLB in PBS-buffer in a mixture of D<sub>2</sub>O/H<sub>2</sub>O with a SLD equal to SiO<sub>2</sub> (red line).

portunity to employ deuteration techniques to highlight molecular components. While deuterated lipids are commercially available, deuterated proteins are expressed in host bacteria in the biochemistry labs of the LMU soft matter group. In order to prepare well defined bilayer samples, a micro fluidic cell has been developed which allows the combination of scattering techniques with fluorescence microscopy (fig. 1).

Instruments at FRM-II which are dedicated to reflectometry studies include N-Rex<sup>+</sup> and REFSANS. REFSANS, which is operated by GKSS, is a timeof-flight reflectometer using a pulsed beam and a 2d detector. To demonstrate the sensitivity of REFSANS to details of the molecular arrangement of lipids in bilayer systems, we have studied the effect of SiO<sub>2</sub> surface charge on the distribution of anionic lipids across a supported bilayer. Note that cellular membranes are highly asymmetric. While anionic lipids such as POPS are common at the inner sheet of the bilayer, their presence at the outer sheet indicates apoptosis, i.e. cell death.

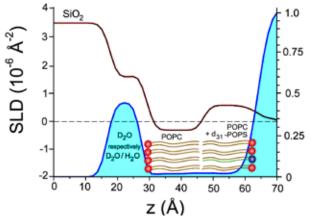


Fig. 3: Calculated SLD profile of substrate and membrane (dark brown curve) and water fraction (blue curve). A schematic of the sample is superimposed.

Here, we use deuterated anionic lipids (d-POPS) mixed with protonated zwitterionic lipids (POPC). Reflectometry experiments (fig. 2) have been conducted by Stefan Stanglmaier and the REFSANS team. The analysis of the neutron data clearly shows that anionic lipids are repulsed from the negative surface charge at the SiO<sub>2</sub> surface (fig. 3). These experiments show that neutron reflectometry experiments at REFASNS are able to monitor asymmetric molecular distributions in lipid bilayers. Similar experiments with membrane proteins associated with Alzheimer and Parkinson disease are planned for the next cycles to come. This initiative is funded by BMBF within the collaborative research project NANOSOFT.

Bert Nickel LMU



## Laboratory for soft condensed matter research

In April 2010 a new research laboratory was inaugurated at FRM II. The main focus of the lab is dedicated to the preparation and analysis of organic colloidal systems. The state-of-the-art research equipment is, however, perfectly suited also for studies on a variety of different samples and scientific questions. The new laboratory is preferentially used for in-house research but is open for studies in cooperation with FRM II too.

For the preparation of colloidal dispersions besides basic equipment, a high pressure homogenizer is available. Dispersions can be prepared at pressures up to 2000 bars and temperatures between room temperature and about 80 °C. Using this device particle sizes below 80 nm can be achieved.

For particle size and shape characterisation a photon correlation spectrometer (PCS), a laser diffractometer (LD) and a Kratky camera for X-ray small angle scattering (SAXS) are available. Particle sizes and size distributions can be determined reliably by these methods within the huge range between 2 nm and 2 mm. The amount and nature of the larger particles can also be studied by a fully motorized light microscope with image analysis and many options as e.g. phase contrast and polarization analysis. Figure 1 shows Sebastian Busch working at the microscope. The PCS is also equipped with a goniometer which can, therefore, allowing static light scattering (SLS) experiments.



Fig. 1: First studies with the research microscope DM6000 (Leica) performed by Sebastian Busch (PhD student).

The phase behaviour of the dispersed nanoparticles can be studied by differential scanning calorimetry (DSC). For this purpose two complementary instruments are available. For high resolution measurements with sample volumes of up to 1 ml in exchangeable sample holders a micro DSC can be used. A picture of the instrument is displayed in figure 2. This instrument also provides the opportunity to operate it in isothermal mode. For faster measurements, which for instance, allow the study of transitions of meta-stable phases a power compensated DSC is used. It allows measurements in the extended temperature range between -170 °C and 750 °C. Due to the power compensation operating mode heating and cooling rates of up to 750 °C can be realized.



Fig. 2: Humphrey Morhenn (PhD student) operating the micro DSC III Evo (SETARAM).

The SAXS instrument (S3-MICROpix, HECUS), already mentioned above and displayed in figure 3, is best suited to be used for liquid and powder samples. Several different sample holders are available including sample holders for pastes and powders, 1 mm capillary, flow through cell, and a system with a rotating capillary. Two different sample stages can be used for temperatures between -20 °C and 300 °C. The Kratky collimator is used in combination with a micro-focus X-ray tube and a point focussing mirror optics. In connection with the lowest background detectors PILATUS 100 K for the SAXS regime (2D) and MYTHEN 1 K for theWAXS regime (1D) a really high performance instrument with a perfect resolution in the SAXS range is available: hardly smeared interferences centered at Q-values as low as Q = 0.012 Å<sup>-1</sup> can be measured nicely and be quantitatively evaluated.

Tobias Unruh FRM II

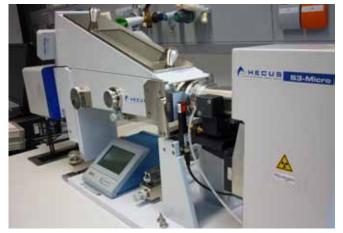


Fig. 3: Photograph of the S3-MICROpix SAXS camera (HECUS, Graz) in the new laboratory for soft matter research at FRM II.





## Guiding the beams: The neutron optics group



Fig. 1: The neutron optics group with head Gunther Borchert (4th from the left) next to the HELIOS facility.

Cutting the glass precisely, sputtering it with a large number of nickel and titanium bilayers to produce a "super mirror", bonding the pieces of glass – recently the neutron optics group at the FRM II has succeeded in performing the whole process of neutron guide production inhouse. The equipment in a new laboratory compasses a precision glass saw, three bonding tables and an x-ray facility to characterize the sputtered layers. The first meters of inhouse produced guides have been accomplished and will replace damaged elements of neutron guides in the neutron guide hall.



Fig. 2: Reflections in a 2m element.

In addition to the fabrication of neutron guides together with the investigation of novel focussing devices, also the design of their shielding, mounting and their precision adjustment at the beam lines and the production of polarized helium-3 for the instruments are duties and responsibilities of the nine members of the neutron optics group. The newest project: a collaboration with international partners to develop more efficient crystals, diamonds, for neutron diffraction and monochromatization.

The quality of the inhouse neutron guides production has reached high international standards. Having started with m = 2 mirrors, the state of the art is now at m = 3.5 (see FRM II news N° 2; p. 6).

Before the super mirrors are assembled to form the neutron guides, they are characterized at the neutron optics group's own instrument TREFF at the FRM II. A probational glass gets sputtered together with the super mirror. This glass is then examined at TREFF, to measure the quality of sputtering and the reflectivity.

Mounting the neutron guides at their destination, they have to be adjusted with a precision of up to 10 micrometres at their position with a directional precision of about 10 arcsec in the guide hall as well as in the experimental hall. In addition, the neutron optics group is engaged to help with the adjustment of forthcoming instruments.

HELIOS, the optical pumping facility, provides polarized <sup>3</sup>He gas for neutron polarization analysis at the FRM II. The straight-line passage of a neutron beam through a <sup>3</sup>He gas with no change of a neutron trajectory enables one to measure a neutron polarization for nearly any divergent scattered beam. It makes <sup>3</sup>He



cells extremely useful in such applications as neutron imaging with polarization analysis, small-angle neutron scattering, offspecular reflectometry and large solid angle polarization analysis. Very promising results have been recently obtained at the instrument ANTARES at FRM II that show this technique to be highly efficient in the study of inhomogeneous ferromagnets. The very high spin-dependent neutron absorption efficiency over a wide range of neutron energies can also be used for polarization analysis of hot neutrons.

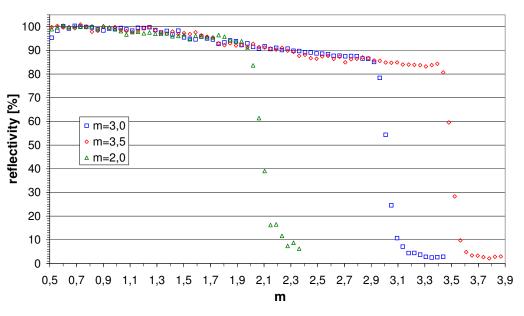


Fig. 4: Reflectivities of three typical super mirrors (up to m = 3.5) measured at TREFF. The super mirrors were produced with the inhouse sputtering facility.



Fig. 3: Josef Weber (left) and Eberhard Kahle (right) prepare a newly sputtered glass for a neutron guide on the bonding table.

This was demonstrated at the instrument POLI-HEiDi where the cells with a polarized <sup>3</sup>He gas were utilised for spherical polarization analysis with the neutrons of the wavelength 0.087 nm.

About 125 cells have been polarized in the last year. Most of them were used at the instruments ANTARES, N-Rex<sup>+</sup>, HEiDi and MIRA. The group is looking forward to a new project with the aim to expand use of polarized <sup>3</sup>He gas.

"Using another technique, we plan an online polarization of <sup>3</sup>He gas directly at the beamline", says Gunther Borchert. Of course, this will require individual facilities for each instrument using <sup>3</sup>He gas.

The promising new project of the neutron optics group could improve neutron flux of some instruments at the FRM II by a factor of 4. In a collaboration between the FRM II, the ILL Grenoble and the HZB Berlin large diamond crystals, which are known to be ideal monochromators for neutron scattering are being developed by the Augsburg University. As the techniques already allow for diamond sizes of 1 cm<sup>3</sup> and 2 mm thickness, the prospects for even larger crystals are high. The goal is to have diamonds large enough within the next two years. For appropriate instruments they will have up to 4 times higher reflectivity than state of the art monochromators. This means an actual amplification of the neutron flux at these instruments of about 4. The project is funded by the German Ministry for Science and Education (BMBF).



some steps beyond simple

What were your duties as

head of the division reactor

enhancement during the last

Before I took over the duties

as head of the department re-

actor enhancement I was al-

ready involved in projects like

cooling systems, the licensing

of the tumour therapy facility

MEDAPP or the integration of new buildings into the li-

censed nuclear facility FRM II.

explanations.

three years?



## "Safe operation is my foremost goal"

#### Interview with the new Technical Director of the FRM II, Anton Kastenmüller

Since April 1<sup>st</sup>, Anton Kastenmüller is the new Technical Director of the neutron source Heinz Maier-Leibnitz. He succeeds Ingo Neuhaus, who took office at the nuclear power plant in Krümmel.

*Mr.* Kastenmüller, you studied at the Technische Universität München, did your Ph.D. here in nuclear physics and you have worked for the FRM II since 2001.

I did my Ph.D. in the field of nuclear physics with the setup of a large spectrometer at GSI Darmstadt. After one year as

postdoc I left this experiment because the setup was nearly finished and started at the FRM II together with a former colleague to establish the detector and electronics lab. Mid of 2001, this was a chance and a risk at the same time because the FRM II was still a construction site and nobody knew, whether this reactor would ever receive an operation license. On the other hand it was very interesting to join the team already years before the nuclear startup.

# Did you ever perform neutron experiments at the FRM II yourself?

During the nuclear startup one task of the detector lab was to measure the neutron flux and the beam characteristics at the neutron guides and the beam tubes. So I did some "neutron experiments" at the FRM II, but no neutron scattering experiments driven by questions in the field of solide state or soft matter physics.

# You have two little daughters. How do you explain your work to them?

Julia is 6 and Lisa 2 years old. Julia is very interested in everything that sounds like science and she has already visited the reactor several times. Therefore she knows a lot about how a reactor works, what happens in the reactor core, what kind of radiation we produce and what the penetration probabilities of neutrons are. She has also visited the control room and since that time she has much fun to start and operate the simple FRM II simulation from the KTG (www.ktg-sachsen. de/NuclearReactor\_FRM2.zip). So we are already



The reactor enhancement department covers handling of all modifications of the reactor and its systems, the coordination of the routine

tor and its systems, the coordination of the routine checks, new projects and the operation of our workshops.

#### What are your goals as Technical Director?

The safe and reliable operation of the neutron source is my foremost goal and of the team of the reactor operation division. A key task is to establish general conditions that allow us to reach this goal. Besides there are some challenging projects in the next years: new buildings, the production facility for Mo-99, the ultracold neutron source, new instruments, etc. We expect an increasing number of scientific and industrial users of the neutron source and have to facilitate successful internationally renowned research projects.

# What is the most promising project within your new responsibility?

As mentioned above the FRM II has started a project for the production of the radiopharmaceutical isotope Mo-99 that is used in medical diagnostics and cancer therapy. Most of the reactors that produce this isotope in Europe are pretty old and there is already a lack of Mo-99 on the market. The FRM II has the perspective to install a production facility near the core that is able to produce a significant fraction of the European demand of Mo-99. The challenge is to realize this project not only from the technical point of view, but also to obtain the necessary nuclear license for the installation and operation of the facility.

Andrea Voit FRM II

## Inside



### **Newly Arrived**

#### **Julia Repper**



What are you doing at the FRM II?

I am second beam line scientist at MIRA and at the spin-echo instrument RESEDA.

What have you done before? I did my PhD at the materials science beam line STRESS-SPEC at FRM II. The subject of my thesis was the inves-

tigation of residual stresses on different length scales in superalloys.

What are your special scientific interests? Materials science and scattering methods in all combinations!

#### **Contact:**

Phone: +49.(0)89.289.14668 Email: julia.repper@frm2.tum.de What are you doing at the FRM II?

I am the second instrument scientist at KWS-1 of the JCNS.

What have you done before? I was a PhD student at the Physics Department, "Lehrstuhl für Funktionelle Materialien" of the Technische Universität München. My re-



search focused on the structural changes in diblock copolymer thin films during solvent vapor treatment.

What are your special scientific interests? Diblock copolymer and microemulsion.

#### **Contact:**

Phone: +49.(0)89.289.10758 Email: **z.di@fz-juelich.de** 

# What are you doing at the

*FRM II?* I am second instrument scientist at the imaging beam line ANTARES.

What have you done before?

In my PhD thesis at FRM II I worked on the investigation of weakly ferromagnetic substances by radiography with polarized neutrons.

#### What are your special scientific interests?

I will continue my work on polarized neutron imaging and perform research on ferromagnetic substances near quantum criticality. Further interest lies in the development of other novel imaging techniques and their scientific application.

#### **Contact:**

Phone: +49.(0)89.289.14718 Email: michael.schulz@frm2.tum.de

#### **Michael Schulz**



#### Michaela Zamponi



What are you doing at the FRM II? I am co-instrument scientist for J-NSE.

What have you done before? I worked for JCNS at SNS in Oak Ridge, USA.

What are your special scientific interests? Soft matter dynamics, in particular polymer dynamics.

#### **Contact:**

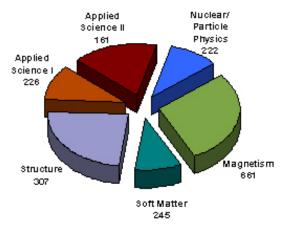
Phone: +49.(0)89.289.10793 Email: **m.zamponi@fz-juelich.de** 

#### Zhenyu Di



## Increase in users at the FRM II

Without any fuss the FRM II went into its sixth year of user operation in 2010. Starting with the first Call for Proposals on November 1st, 2004, the first users visited FRM II when the routine operation started on March 3rd, 2005. Since then more than 1000 proposals have received more than 7000 days of beam time at the instruments of FRM II. Measurements have been performed in all areas of science and have been published in high impact journals such as Science, Nature Physics, Physical Review Letters and Biophysical



Beam days requested at the FRM II instruments in 2010, proposal round 11.

*Journal*. In 2009 about 1000 scientific visitors came for experiments to the FRM II.

At the last proposal round at FRM II and JCNS on January 29<sup>th</sup>, 2010, a total of more than 300 proposals were submitted, asking for about 2500 days of beam time. This led to an overall overbooking factor for the available instruments of 2. The majority of users tradi-

## **Call for Proposals**

#### Next deadline is October 8th, 2010

Submission of proposals to instruments at FRM II and JCNS can be done at the digital user offices

#### user.frm2.tum.de fzj.frm2.tum.de

The Call for Proposals can be downloaded from

www.frm2.tum.de/en/user-office/news-dates Details of the instruments and sample environment available can be obtained at

www.frm2.tum.de/en/science

The review of the proposals will take place between November 26<sup>th</sup> and 30<sup>th</sup>, 2010. Results of the reviews will be online about two weeks later. The experiments of the accepted proposals will cover the reactor cycle 26 (March to May 2011) and cycle 27 (June to August 2011).

**Please note** that due to the refurbishment of the positron source at FRM II the reactor will be shut down between October 16<sup>th</sup>, 2010, and end of February 2011. Unfortunately this will slightly reduce the possible beam days for experiments in 2011 by 15 %.

tionally come from German universities and research centres, however more than 10 % of the proposals came from outside Europe including China, Korea, Australia, Brazil and Argentina. Most of the European users visiting FRM II and JCNS received financial support by the current running FP7 NMI3 program.

The FRM II recognizes the increasing number of proposals and scientific visitors since start of the user operation five years ago as a clear vote of acceptance and acknowledgement of

the performance of the facility and its instruments. We aim to improve further our performance with new developments and projects as the new MIRA2, Poli-Heidi, the upcoming new instruments MARIA, SANS-1 or the BioDiffractometer. All these projects and instruments offer new opportunities for science with neutrons at the FRM II.

Thomas Gutberlet JCNS

## **3rd FRM II User Meeting**

Before the FRM II will have a longer shut down period between October 2010 and February 2011, the next FRM II User meeting will take place on October 15<sup>th</sup>, 2010, at the Physics Department of TU München at Garching.

We hope to welcome a large number of our users which we encourage to submit their work for oral or poster presentation at the FRM II User Meeting registration web page

#### www.frm2.tum.de/user-meeting-2010



The one day meeting will consist of oral presentations of scientific work achieved by the FRM II and JCNS user community with an extended late afternoon poster session in combination with traditional Bavarian food and beer. Registration for the FRM II User Meeting is free of charge.

# **User Office**



# Upcoming

#### July 11-13, 2010

JCNS Workshop Modern Trends in Production and Applications of Polarized <sup>3</sup>He (Ismaning, Germany) www.jcns.info/Workshop 3He

September 6-17, 2010 14th JCNS Laboratory Course - Neutron Scattering (Jülich/Garching, Germany) www.jcns.info/wns lab now/

September 26-29, 2010 International Workshop "Neutrons for Global Energy Solutions" (Bonn, Germany) www.fz-juelich.de/iff/nges2010/

#### September 29-1, 2010

6<sup>th</sup> International Workshop on Sample Environment at Neutron Scattering Facilities (Hersching, Germany) www.frm2.tum.de/en/aktuelles/events/se-workshop

#### October 4-8, 2010

JCNS Workshop "Trends and Perspectives in Neutron Scattering: Magnetism and Correlated Electron Systems" (Bernried, Germany) www.jcns.info/Workshop\_Magnetism/

October 15, 2010 3rd FRM II User Meeting (Garching, Germany) www.frm2.tum.de/user-meeting-2010

October 21-23, 2010 SENSE 2010 - Superconductivity explored by Neutron Scattering Experiments (Grenoble, France) www.ill.eu/news-events/events/sense2010



**JCNS (N° 8)** fzj.frm2.tum.de

## IMPRINT

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#### Latest news:

# Race against time: Fast restart after motor failure

The operation of the FRM II neutron source was unexpectedly intermitted on June 17th. The reason was a defect motor of the compressor for the cold source,



automatically shutting down the cold source and by this the entire reactor. The race against time began. During the weekend, the electrical engineering division found an identical motor in Berlin and guickly transported it to Garching. This is extraordinary because comparable engines (500 kW, 2.5 tons) have

long delivery times. The new engine was adapted and then built in and mechanically adjusted within one day. Thanks to the great effort and extra working hours of the FRM II workshop team, cold source group, external companies, the exchange was accomplished within only one week. The cycle is continued since June 25th and will be prolongated till July 24th.

The next cycle will start as scheduled on August 17<sup>th</sup>.

Andrea Voit FRM II

