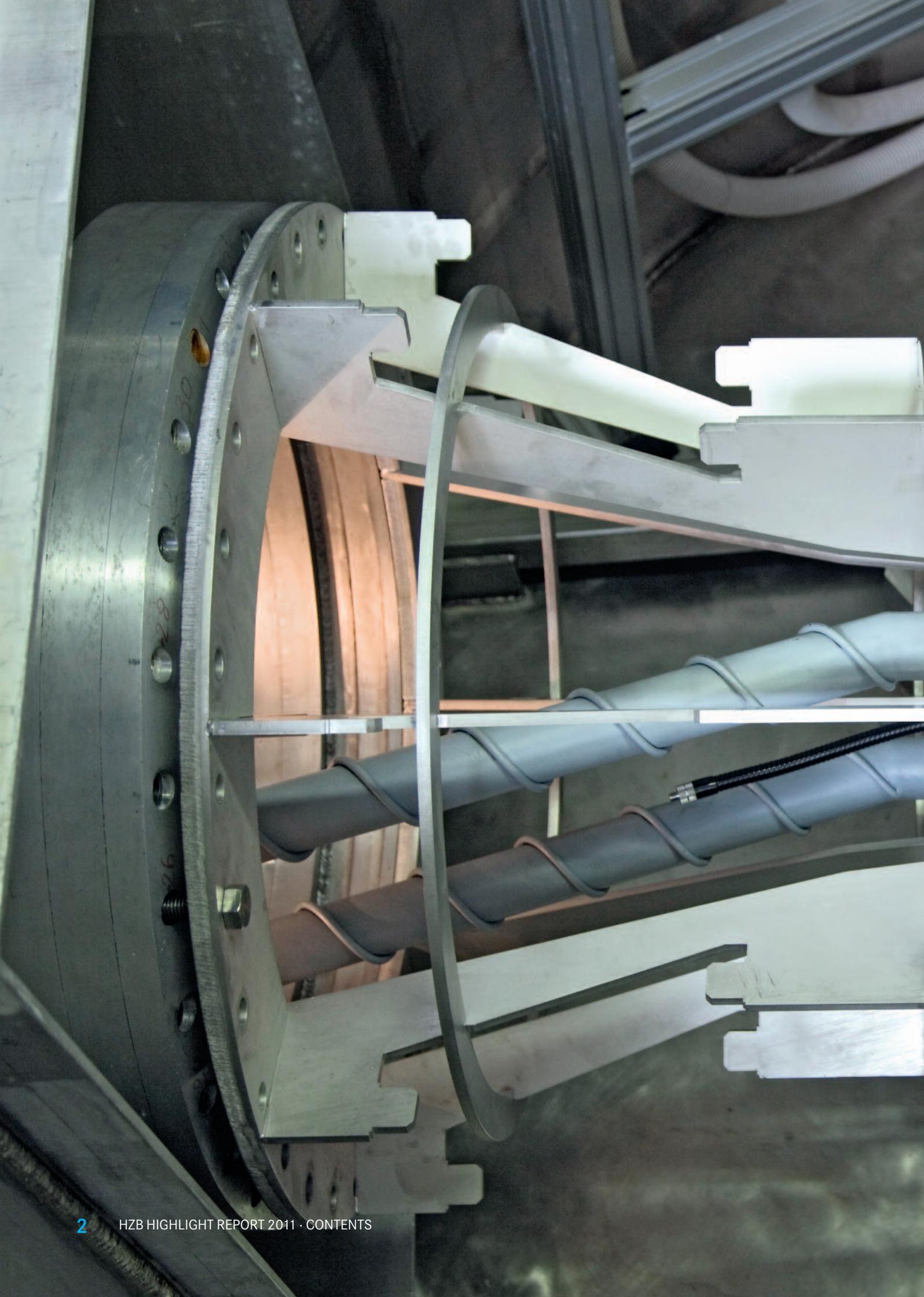


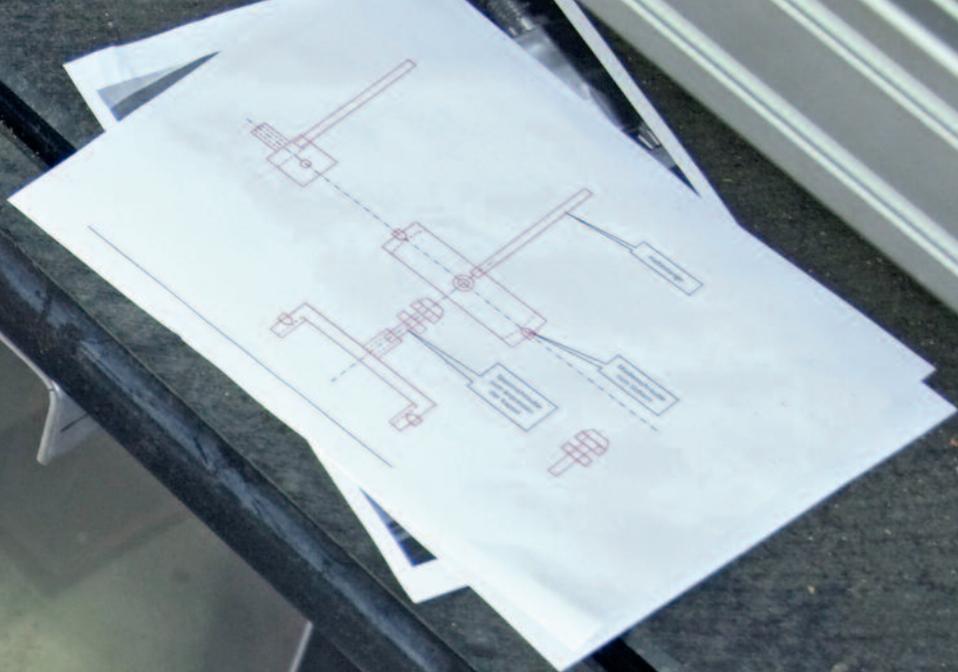
BUNDLING ENERGY REALIZING VISIONS



# HIGHLIGHTS 2011

Annual report with research highlights at the  
Helmholtz-Zentrum Berlin für Materialien und Energie





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## SHAPING THE FUTURE OF RESEARCH

In many ways 2011 was a very special year for the Helmholtz-Zentrum Berlin für Materialien und Energie. With upgrade of the research neutron source BER II and the successful start of our future-oriented BESSY II projects, we were able to pave the way for many important future projects. One of them centres on setting up dedicated measuring stations for photovoltaic research at the BESSY II storage ring.

Photovoltaic research has gained impetus not least as a result of the energy policy change in Germany, and a rising interest in this field of research is also noticeable on an international level. At HZB, we have the unique possibility of making it easier to conduct photovoltaic research using our large-scale research facilities. Within the scope of our planned large-scale project *EMIL* and together with the Max Planck Society, HZB will create an infrastructure at BESSY II over the coming years that is unique in the world to permit in-situ investigations on materials for converting and storing energy. *EMIL* stands for Energy Materials In-Situ Laboratory Berlin. The investments made as part of the *EMIL* project will succeed in significantly expanding the research opportunities at the synchrotron source BESSY II in Berlin-Adlershof. An external committee of experts brought in by the scientific advisory board submitted an extremely positive evaluation of the *EMIL* project in September 2011 and strongly recommended its realisation. The supervisory board of HZB then gave the go-ahead for construction of *EMIL*. The BMBF (German Federal Ministry for Education and Research) and Helmholtz Association are supporting the ambitious project.

We have also made a successful start with our future-oriented project *BERLinPro*. We are using a feasibility study to check whether the principle of a linear accelerator with energy recovery (Energy Recovery Linac) is fundamentally possible. The first photoelectrons were accelerated for this purpose in April 2011 at HZB with a superconducting electron source (SRF gun). This was the first time that an electron beam was generated using a superconducting



Prof. Dr. Anke Kaysser-Pyzalla and Thomas Frederking

radio-frequency photoinjector from a superconducting photocathode. We have thus come a big step closer to our goal of realising an Energy Recovery Linac, because for such an accelerator, one needs electron sources of maximum brightness.

A particular focus in 2011 was to upgrade our BER II neutron source. We started work in the autumn of 2010 and completed the project at the end of March 2012. You will find a detailed report on the following pages. We were extremely pleased to note a marked improvement in the neutron source after recommissioning the BER II, brought about by the exchange and technical refinement of important components. This was particularly pleasing because a public discussion about the facility started during the extensive conversion work. In dialogue with members of the public and also with the media, we conducted the discussion with great openness and transparency. We were able to turn the increased interest to account and also to make the research conducted with BER II tangible for all interested parties. In September 2011, the nuclear supervisory authority certified a high degree of robustness for the BER II. The mandated stress test was passed with flying colours. Since the spring of 2012, the facility has been up and running again and it will make an indispensable contribution to research for many years to come.

Nevertheless, it is spallation neutron sources that are likely to be the research hot topics of the future. This makes it necessary for us to get a head-start in designing and building instruments for future spallation neutron sources. The Helmholtz-Zentrum Berlin is contributing actively to the project for building the European Spallation Neutron Source (ESS) in Lund, Sweden. A measure for the significance of this project – in which 17 European nations are involved – is the estimated 1.48 billion euros total costs for planning, construction and operation of the ESS. Construction will begin in 2013 and it is estimated that the ESS will be available for researchers from all over the world in the 2020s. HZB experts are involved in this future European neutron source in terms of the building concept as well as development of the requisite instruments.

In this Highlight Report, you will find information about the start of our “Campus 2030” project, which the Helmholtz Association is sponsoring as a model project. For it is not only the technical facilities that HZB is making efforts to keep bang up to date, but also the entire infrastructure. Within the framework of this project, the aim is to develop ideas for the campus of the future which are also attractive for other locations. The Lise-Meitner Campus in Wannsee, for example, is absolutely ideal for this. This campus is an innovative hub for science and also an international meeting place, but some of the buildings hailing from the 1950s display a glaring need for renovation. It is thus perfect for a model project, whereby besides the energy supply and building renovation, the objective is also better public transport links and transparent communication paths on the campus. Thanks to the manageable size of the Lise-Meitner Campus, the chances are good that the results of “Campus 2030” will be realised in the medium and long term.

But more important than buildings and technology are the people who work at HZB. We were therefore delighted to register the numerous prizes awarded to HZB staff members in 2011. For example, Prof. Dr. Emad Flear Aziz, head

of the young investigator group for the structure and dynamics of functional materials. We congratulate him on being awarded the ERC Starting Grant, with which his research project will be funded over the next five years. Particularly noteworthy are also the joint appointments for important chairs that HZB has realised together with the universities of the Berlin and Brandenburg region. Prof. Dr. Susan Schorr, Prof. Dr. Joachim Dzubiella and Prof. Dr. Alexander Matveenko – all appointed to the FU Berlin or the Berlin Humboldt University in 2011 – are prime examples of just how closely the HZB cooperates with the universities and just how well the extramural research of HZB is networked with university research and regional education. Such appointments are at the same time important career stages for the scientists themselves.

There have also been two changes in the HZB management: Prof. Dr. Wolfgang Eberhardt, director for more than seven years at BESSY and almost three years for the energy business segment at HZB, returned to research in mid-2011 and is now active at DESY in Hamburg. And Dr. Ulrich Breuer, commercial director at HZB, moved to the Karlsruhe Institute for Technology (KIT) on 1 January 2012 and assumed the function there of vice-president for finance and economics. Our thanks and appreciation are due to both former HZB directors along with our very best wishes for their future.

The many excellent scientific reports published in 2011 once again constitute an indicator of the unbroken high quality of research at HZB. They were written not only by external users of our large-scale research facilities but also by HZB staff members. Exemplary cases here are the nanomagnet research and the structure analysis of protein crystals as well as the investigation of materials for thin-film solar cells. Deeper insights into these and many other exciting results of research conducted at HZB can be found in this HZB Highlight Report 2011. They all verify the sustained attractiveness of HZB as a major centre for scientific research.

  
Prof. Dr. A. Kaysser-Pyzalla

  
Thomas Frederking

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## NEUTRONS FOR RESEARCH – BER II SUCCESSFULLY UPGRADED

After an upgrade period lasting almost one and a half years, HZB put the neutron source BER II back into operation again on 27 March 2012. Prior to this, the BER II passed the special stress test mandated by the Berlin Senate. Researchers and scientists now once again have **excellent conditions for conducting experiments**. The research location has become even more attractive since the upgrade.

**2** October 2010; 9 p.m. A view of HZB's reactor hall in Wannsee. The control rods are lowered into the core of the neutron source BER II. They interrupt the chain reaction that drives the reactor and delivers valuable neutrons for research. Although the operator teams shut the reactor down at least every three weeks for maintenance and routine inspection, this shut-down is a special one. The BER II remains shut down initially for several months and in the end for one and a half years. But why?

Not only the neutron source but also the neighbouring experiment halls required extensive maintenance and modernisation. The conical beam tube – one of the most important reactor components – needed exchanging. It encompasses the cold neutron source (CNS) in which the neutrons coming from the reactor core are braked in super-cooled hydrogen gas. The moderator cell through which the neutrons rush into the neutron guides is the central

component thereby. Just as the conical beam tube, both these components needed to be removed and remounted in a technically improved form to enable a more intensive neutron flow. Some 50 percent more neutrons will exit the new moderator cell as opposed to the old one. The neutron guides through which the neutrons are conveyed to the experiments were also renewed. They received a supermirror coating which ensures that even more neutrons arrive at the experiment stations, some of which the researchers still want to modernise. The project facing HZB in the autumn of 2010 was nothing if not ambitious.

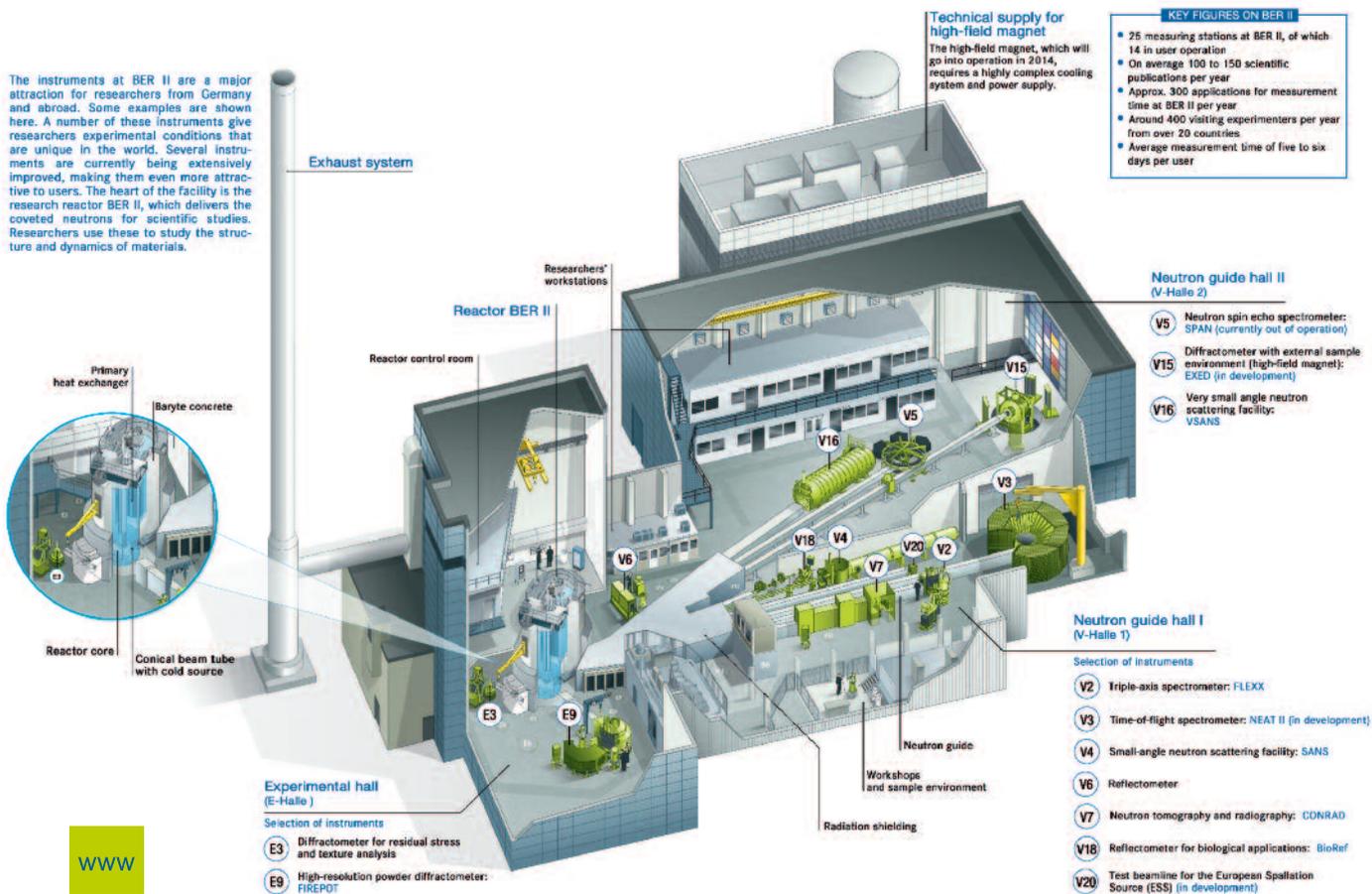
### New components for BER II

The reason: the reactor components had provided reliable service for around 20 years. “Over time, however, the metal that is subjected to constant neutron bombardment becomes brittle,” says Stephan Welzel, who planned and

was responsible for the work on the reactor upgrade in his capacity as chief coordinator. “We were able using comparison materials installed at other locations in the reactor to calculate in advance just how quickly the parts age.” Based on this, it was clear as early as 2006 that the conical beam tube and the cold source would need exchanging in 2011 at the latest. “If you work with radioactive material, you naturally don't just wait until something breaks down,” says Welzel. Exchanging the conical beam tube was



The conical beam tube is brought into position.



therefore not a repair but rather a precautionary maintenance measure. But one that nevertheless demanded an enormous amount of planning and coordination. In view of this, the scientists had erected a mock-up a good one and a half years prior to servicing in order to practise every single manual action and every machine operation. Once the reactor was shut down in October 2010, work could begin. "By the middle of March 2011, we had removed the conical beam tube and the cold source, dismantled and packed them and then handed them over for final disposal to the central agency for light- and medium-radioactive waste," describes Stephan Welzel the first but crucial work step. Delays set in, as Welzel relates: "With a project such as the reactor upgrade, delays are almost inevitable because on the one hand, there is no standard procedure for such a complex project, and on the other hand, every step must be carried out in strict compliance with maximum safety and quality demands."

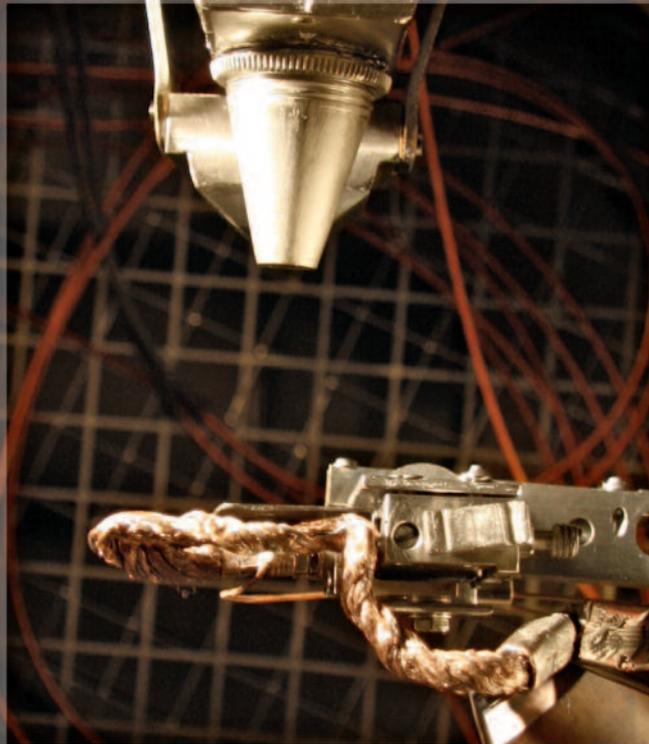
### The cold source fulfils highest safety standards

Although the new cold source could have been installed in April 2011, this did not happen until 6 months later. Stephan Welzel says: "The forged material must meet the very highest requirements, and it is only produced in small amounts. And in spite of us having an extremely long lead time, the alloy was not available in the required quantity on the market." There was no alternative but to wait: "Safety is top priority," says Welzel: "We may only use approved and certified construction materials for the reactor." Despite this, there was no standstill as such. And within the

scope of the upgrade, the entire neutron guide system that supplies the neutrons for the experiments in neutron guide hall I was remodelled. "Instead of five neutron guides as before, the researchers now have six of them available for experiments," says Thomas Krist, who was responsible for rebuilding the neutron guides. Three of them were also doubled in width. Krist: "The new guides have a supermirror coating that improves the neutron flow." To this end, all five neutron guides had to be completely dismantled and reassembled using a new geometry to six neutron guides. In November 2011, the cold source was finally delivered. "On the very day that the supervisory board of HZB had a meeting," remembers Stephan Welzel: "I was so glad that I was able to give Mrs Kaysser-Pyzalla, the scientific director, the good news." The project now gained momentum again: the cold source was installed in the new conical beam tube in early 2012.

27 March 2012. A look into HZB's reactor hall in Wannsee. The control rods are raised out of the core of the BER II neutron source. The chain reaction starts up. "The material that we built into the new conical beam tube, the moderator cell and the cold source underwent extensive trials and tests. We passed all tests with flying colours," says Stephan Welzel: "But it is still stipulated in spite of this that we start up the reactor gradually to enable us to keep a close eye on how the new components behave." It takes six days for the BER II to run up to full power. Since then it has again been delivering a precious resource for which several hundred scientists travel to Wannsee: neutrons for research.

*hs/co-author: Annick Eimer*



# HIGHLIGHTS FROM USER EXPERIMENTS

In 2011, the neutron source BER II in Berlin-Wannsee was unavailable to the many scientists from around the world who normally work at its measuring stations. This was owing to renovations that started in October 2010 and were completed in the spring of 2012. With the new conical beam tube, an optimised moderator cell and cold source, and the updated and significantly improved neutron guides, researchers have had excellent conditions for their neutron experiments since the restart of BER II in spring 2012.

While these renovations were going on, most of the research at HZB in 2011 was concentrated instead at the electron storage ring BESSY II in Berlin-Adlershof. Scientists from the Center for Nanointegration at the University of Duisburg-Essen (CENIDE), for example, used the high-brilliance X-ray beam to study nanomagnets as are now used in numerous medical and technical applications. They discovered how to manufacture these tiny magnets with special properties.

Using the scanning X-ray microscope MAXYMUS built at HZB in collaboration with the Max Planck Institute for Intelligent Systems, a group of scientists managed to reverse needle-like magnetic structures called vortex cores twenty times faster than ever before. This may offer new routes for stable, fast and highly miniaturised data storage.

Fluorescent proteins are important experimental tools for the biosciences that can be used to study the processes of life inside cells and organisms at the molecular level. Researchers from the Max Planck Institute for Biophysical Chemistry in Göttingen and from FU Berlin working on Beamline 14.2, which is operated in the scope of the Joint Berlin MX-Laboratory, managed for the first time to measure the crystal of a single fluorescent protein in both lit and unlit state. Until now, a given protein crystal could only be studied in one of these two states, and not both.

We invite you to read more about these and many more examples of user experiments at HZB over the following pages.



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## TAILORED NANOMAGNETS

**Magnetic nanoparticles** are extremely versatile. Using instruments at HZB, researchers from the University of Duisburg and Essen have discovered how nanomagnets can be developed for all kinds of applications.

**T**eensy, nanoscale particles have fascinating properties: They are so small that surface effects dominate their physical behaviour and they manifest quantum mechanical phenomena. They can also be effortlessly manoeuvred into obscure places in the human body, meaning they can be used as ferries for drugs or even to treat diseases directly. When nanoparticles have magnetic properties, engineers and medical professionals can easily move or guide them using electromagnetic fields.

### Dwarf helpers in medicine and technology

This is already being done in cancer medicine, for instance, as a means to combat tumours by so-called hyperthermia using nanoparticles. The tiny magnetic particles are transported to the tumour directly by injection – or in future through the blood stream – and then their magnetisation is brought into rotation by a rapidly alternating magnetic field. This heats up and ultimately destroys the tumour. Magnetic nanoparticles also work as a contrast medium to reveal the finest structures of body tissue in MRI tests. And we may find them in tomorrow's high-performance electronic data memories, where magnetic fields will align them in specific directions to mark the digital ones and zeros.



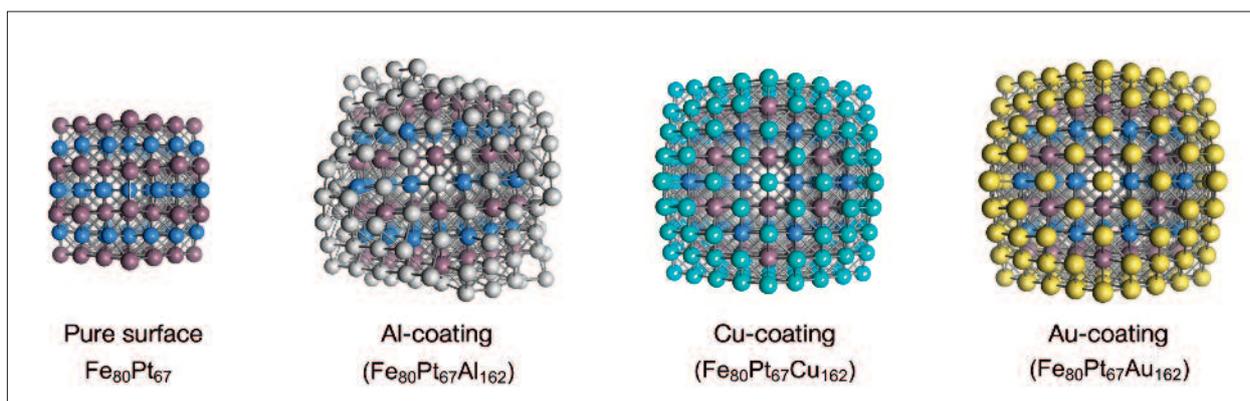
View inside the experimental hall at the electron storage ring BESSY II. The metallic nanoparticles covered with various shells were studied here.

Naturally, each of these specific applications for the versatile particles focuses on different properties, some requiring the complete opposite behaviour to others. In hyperthermia cancer treatment, for example, the nanoparticles have to have easily reversible magnetic alignments for them to generate sufficient destructive heat. In data memory, on the other hand, they have to retain their alignment stably over very long periods of time.

To tweak their magnetic nanoparticles to requirements, researchers have so far been working from experience and from the results of isolated and rather unsystematic experiments. The design and manufacture of these particles is almost like modern alchemy. Now, a group of scientists from the University of Duisburg-Essen has developed a recipe of sorts for tailoring magnetic nanoparticles precisely to the specific targets of different technological and medical applications. The researchers made use of the high-brilliance X-rays from the electron storage ring BESSY II at HZB in developing this recipe for nanocuisine.

### The right shell makes all the difference

For their studies, the researchers focussed on metallic nanoparticles made of an alloy of iron and platinum (FePt) – a material that possesses a pronounced magnetic anisotropy (directional dependence) and is therefore highly suitable for many applications. “We wondered what would happen if we coated these particles with another material,” reports Prof. Heiko Wende, workgroup leader in the experimental physics department and at the Center for Nanointegration at the University of Duisburg-Essen (CENIDE). “Such a shell protects the nanoparticles against oxidation,” the physicist explains. Yet, it also changes their physical and chemical properties, and exactly how it will do so has always been difficult to predict before carrying it out in practice. Heiko Wende and his colleague Dr. Carolin Antoniak used the X-rays generated at BESSY II to take the first detailed and systematic look at the influences of this shell. They measured how strongly different shells absorb various wavelengths of X-ray light. The workgroup of physicist Prof. Michael Farle from Duisburg assisted the researchers, who



For the experiments on metallic nanomagnets, the scientists coated an iron-platinum compound (blue and violet atoms, left) with aluminium (white atoms, mid. left), copper (turquoise atoms, mid. right) and gold (yellow atoms, right). Next, they studied the magnetic properties of the material combinations.

chose gold, copper and aluminium as the material for coating the spherical FePt particles of around six-nanometre (millionths of a millimetre) diameter.

#### Experiment and simulation make a perfect team

Antoniak and Wende compared the results of the experiments at HZB with the results of simulations previously generated by research colleagues Dr. Markus Gruner and Prof. Peter Entel. The theoretical physicists had used the supercomputer JUGENE at Forschungszentrum Jülich – currently one of the fastest and most powerful supercomputers in the world, with a computing power of up to a quadrillion operations per second. On this gigantic machine, Gruner and Entel had used atomic-scale models to simulate how different metallic shells would influence the properties of the iron in the coated nanoparticles.

The results of the X-ray absorption measurements beautifully confirmed the theoretical conclusions from the computer simulations. “The calculations and measurements complemented each other outstandingly and are mutually beneficial,” Heiko Wende proudly announces. While the theoretical calculations had delivered highly precise results, they had namely been based on assumptions. These assumptions have now been experimentally confirmed.

“The combination of theory and practice has delivered us our first precise understanding of the physical processes in nanoparticles,” says Wende – “and thus lays the foundation for reliably predicting the properties of such magnetic nanoparticles in future.”

The scientists working with Wende discovered that an aluminium shell strongly modifies the behaviour of the iron-

platinum particles. If the particles are coated with gold or copper, on the other hand, then their properties hardly change. “Accordingly, a hard magnetic material with a large saturation moment should be coated with copper, for example,” Carolin Antoniak concludes. “For platinum-iron particles of high surface iron content, a gold shell can produce a soft magnetic material that still possesses a large magnetic moment.” Hard magnetic materials can only be changed by an external magnetic field with difficulty. Soft magnetic substances, on the other hand, respond quickly to the action of an external field. Saturation refers to the maximum possible magnetisation a material can obtain inside a magnetic field. This value namely also varies between materials.

The new insights are extremely useful for applications in hyperthermia, for example. And the researchers have come across many other useful correlations that could serve as starting points for developing entirely new nanoparticles with novel properties. Heiko Wende and his research colleagues already have some ideas: One of these is to coat nanomagnets with organic materials instead of metallic ones in future. Wende concludes: “Then we might be able to modify the properties of the particles using light, for example.”

*rb*

Nature Communications, 2: 528, (DOI: 10.1038/ncomms1538):

A guideline for atomistic design and understanding of ultrahard nanomagnets, Carolin Antoniak, Markus E. Gruner, Marina Spasova, Anastasia V. Trunova, Florian M. Römer, Anne Warland, Bernhard Krumme, Kai Fauth, Shouheng Sun, Peter Entel, Michael Farle & Heiko Wende

## MODIFYING GRAPHENE AS REQUIRED

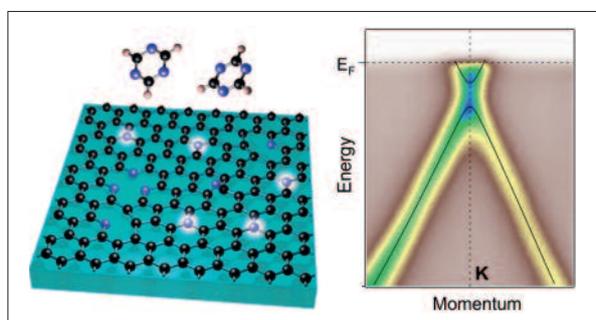
An international research team worked at HZB to study the growth process, structure and electronic properties of **N-graphene**

Ever since its discovery, graphene has been hailed as a wonder material. Even its physical appearance is fascinating. It consists of carbon atoms arranged into a honeycomb structure, similarly to graphite. Graphene, however, is only one layer of atoms thick. A material can't get any thinner than that. Nevertheless, it is highly mechanically and even chemically stable. It is also an extremely good electrical conductor. That makes graphene attractive for future generations of electronic components, such as ultra-fast transistors, for example. Other possible applications are as electrochemical biosensors, inside lithium-ion batteries or inside fuel cells.

Whatever the application, it will always be necessary to adapt the electronic properties of the graphene, especially the energy levels at which the electrons race around the atomic nucleus or through the crystal lattice. Similarly to other semiconductor materials used in electronics, it is advantageous to dope graphene: Incorporating foreign atoms into the carbon lattice puts additional charge carriers into the material that can influence the electronic energy level.

### Nitrogen atom replaces carbon atom

Researchers working with Dr. Dimityr Usachov of the University of St. Petersburg and Dr. Alexander Grüneis of the University of Vienna and IFW Dresden have successfully produced nitrogen-doped graphene (N-graphene). Together with colleagues from Lund, Trieste and HZB, they studied the growth process, structure and electronic properties of this material. "Nitrogen has exactly one electron more than carbon and in principle should integrate well into the carbon crystal lattice," Grüneis explains. Yet, there are various possible configurations it can assume when it does. The ideal is a 1:1 exchange with a carbon atom. Then the nitrogen atom would have three directly neighbouring carbon atoms so that the hexagonal graphite structure of the graphene would remain intact, meaning it would retain its outstanding electrical conductivity as well. However, there are other configurations in which nitrogen will have only two direct carbon neighbours, or in which a pentagonal structure will form instead of the hexagonal structure. These disruptions in the



In nitrogen-doped graphene (left), additional atoms and thus charge carriers are incorporated into the carbon lattice in order to control its electronic properties. In the angle-resolved photoemission spectrum (right), the researchers check whether the nitrogen has in fact donated electrons.

crystal lattice would have negative effects on the electronic properties. To produce their N-graphene, the researchers exposed a nickel crystal to triazine vapour, a compound of carbon, nitrogen and hydrogen. Then they used X-ray photoelectron spectroscopy (XPS), among other methods, to investigate how the graphene layer and its honeycomb structure had formed by this process, and how the nitrogen molecules were incorporated into it. They shone X-ray light of varying energy onto the sample and analysed the energies at which electrons were knocked out of the nitrogen atoms. From this, they could draw conclusions on the binding states of the nitrogen atoms and thus on their surroundings.

### Electronic properties measured

Generally, the researchers discovered, N-graphene grows similarly to undoped graphene, although at a slower rate. The nitrogen atoms make up only a tiny fraction of about a half to two percent of the atoms in the crystal lattice. "We found that they are incorporated into the crystal lattice in various electronically unfavourable configurations at first," Alexander Grüneis reports. "Bringing them into the desired configuration requires thermal post-treatment." At this stage, they have to loosen the strong coupling of the graphene onto the underlying nickel crystal. For this, the researchers apply a known trick: They vapour-deposit a layer of gold that pushes

its way under the graphene layer to form an intermediate layer. The graphene is then quasi detached. Next, it is heated to 500 degrees Celsius. When this is done, around 80 percent of the nitrogen rearranges into the desired configuration.

The researchers confirmed this by measuring the electronic properties. For this, they used angle-resolved photoemission, where they analysed the angle at which the X-rays knocked out the electrons. “For our measurements, we used the especially bright radiation from BESSY II at the beamlines of UE52-PGM, RBGL and 1<sup>2</sup> ARPES,” Grüneis says. “Its advantages are its adjustable energy, for determining the various possible electron levels, its highly monochromatic light, which delivers very clear measurement results, and its par-

ticularly high sensitivity.” The last is essential for being able to measure the tiny amounts of nitrogen in the first place. The results show that the nitrogen, when it is incorporated into the graphitic configuration, acts as an electron donor as planned, so that the electronic properties can be modified in a targeted manner. “These findings are an important basis for the future use of graphene in electronics,” Grüneis resumes. ud

Nano Letters, 2011, 11 (12), 5401–5407 (DOI: 10.1021/nl2031037): Nitrogen-Doped Graphene: Efficient Growth, Structure, and Electronic Properties, D. Usachov, O. Vilkov, A. Grüneis, D. Haberer, A. Federov, V.K. Adamchuk, A.B. Preobrajenski, P. Dudin, A. Barinov, M. Oehzelt, C. Laubschat, D.V. Vyalikh

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## IRON – A CONTRADICTIONARY SUPERCONDUCTOR

Iron-based superconductors exhibit some very strange properties. An international team of researchers working together with the **HZB research group PANDA** in Garching near Munich have come across a previously unknown effect in the formation of the superconducting phase.

In 2006, Japanese scientists discovered a surprising new class of superconductors. Unlike the high-temperature superconductors known since the 1980s, these materials are based not on copper compounds, but on a lattice of iron and arsenic atoms instead. This discovery raised hopes for a better understanding of the origin of high-temperature superconductivity – and for developing innovative and lucrative applications for it. Yet, to reach this goal, researchers still have to resolve a number of questions regarding the properties of these iron-based ceramics. Take for instance a recently discovered and little understood effect: In the inelastic scattering of neutrons at the crystal lattice of these superconductive materials, a distinctive excitation shows up – an increased scattering rate for specific energy and momentum transfers. This unusual effect is only observed in the superconducting phase so, logically, it must be associated with its formation. Solving this mystery could help understand how iron-based superconductors form and how to produce them in a targeted manner for applications. “Previous understandings suggest that the energy-dependence and the type of excitation ought to change in a certain way when an external magnetic field is applied,” says Enrico Faulhaber, second in command of the instrument in the joint research group PANDA/TU Dresden at the re-

search neutron source Heinz-Meier-Leibnitz (FRM II) in Garching near Munich. Faulhaber was a cooperation partner of an international research team from China, France, Germany and the USA that conducted experiments in Garching in 2011.

### Superconductors change the scattering

They set out with their experiments to find a physical explanation for the puzzling neutron scattering. Some physicists believe it to be a result of electron spin excitation (spin is a quantum mechanical property often explained for simplicity as the particle’s rotation about its own axis). To test this hypothesis, the researchers studied two different iron-based superconductors ( $\text{FeSe}_{0.4}\text{Te}_{0.6}$  and  $\text{BaFe}_{1.9}\text{Ni}_{0.1}\text{As}_2$ ) that become superconductive at temperatures below 14 and 20 kelvin (minus 259 and minus 253 degrees Celsius), respectively. They placed the samples in a strong magnetic field and systematically studied the inelastic scattering of slow neutrons. “The equipment we used for this is special for the extremely strong magnetic field of up to 14.5 tesla that can be applied during the scattering experiment,” explains Faulhaber, “and the high resolution of the PANDA instrument – a triple-axis spectrometer – combined with the intense neutron flow from the cold source



The cold neutron three-axis spectrometer PANDA operated by HZB at the Heinz-Meier-Leibnitz research neutron source (FRM II) in Garching near Munich was used for studying two iron-based superconductors.

### New theoretical models needed

This finding makes the phenomenon even more puzzling to the physicists. “While we did confirm that the scattering characteristics and the appearance of superconductivity are correlated,” says Enrico Faulhaber, “we now have an additional effect that we don’t understand.” And this appears to be universal, since it can be seen in various iron-based superconductors. The physical causes for the distinctive scattering are – for the moment – still a mystery. The phenomenon must be explained, since it is important for understanding the microscopic processes of high-temperature superconductivity. Further experiments on larger and specially prepared samples and – for improving the statistics – over an even longer period could improve the data basis and thus perhaps help to develop an explanation. Yet, for Enrico Faulhaber, the ball is now equally in the theoretical physicists’ court: “They first have to develop suitable models that plausibly describe the behaviour of iron-based superconductors,” he believes.

of FRM II, where the instrument is operated.” Because the scattering at the samples is altogether weak, these measurements had to be performed over a relatively long period. Together with previous experiments at other large-scale research facilities, a consistent picture has emerged: Contrary to expectations – and contrary to the behaviour at the critical temperature where superconductivity breaks down in the material – the strength and nature of excitation remained largely unaffected by the magnetic field. This applied to both samples, even though their structures were different.

Working from these models, the experimental stations at HZB could then be used again to put the theoretical concepts to the test in further experiments. The nature of superconductivity in iron arsenide-based substances remains a thriller – and a scientific “hot iron”. *rb*

Phys. Rev. B 84, 024518, 2011 (DOI: 10.1103/PhysRevB.84.024518): Effect of the in-plane magnetic field on the neutron spin resonance in optimally doped  $\text{FeSe}_{0.4}\text{Te}_{0.6}$  and  $\text{BaFe}_{1.9}\text{Ni}_{0.1}\text{As}_2$  superconductors, S. Li, X. Lu, M. Wang, H. Luo, M. Wang, C. Zhang, E. Faulhaber, L.-P. Regnault, D. Singh & P. Dai

## A QUICK SWITCH

Miniscule **magnetic “needles”** are ideal components for a new generation of data memories. Researchers from the Max Planck Institute have now discovered how data can be processed with extreme speed in such memories.

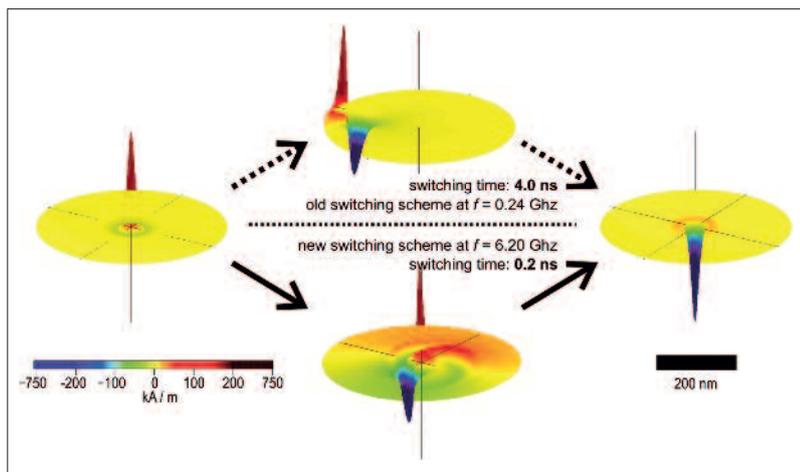
Materials used for storing data have to satisfy two requirements: They have to store digital information over a long period of time, and they have to allow this data to be rapidly and easily stored, modified or deleted. Candidates for future powerful memories that fulfil these requirements are the “magnetic vortex cores”. “These are nanoscale, swirling structures that arise on tiny platelets made of ferromagnetic material,” Dr. Matthias Kammerer explains. The physicist, who researches in the workgroup of Dr. Hermann Stoll at the Max Planck Institute for Intelligent Systems (MPI-IS, formerly the Max Planck Institute for Metals Research) in Stuttgart, studied such formations for his doctoral thesis. He came across a mechanism that allows extremely fast switching of the cores between two different states.

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For his experiments, the physicist used the X-ray microscope MAXYMUS that researchers from MPI-IS had built under the direction of Prof. Gisela Schütz, together with experts from HZB. It has been in operation at HZB since the end of 2009. Working together with researchers from the Universities of Gent and Regensburg, Kammerer examined tiny discs of permalloy – an alloy of nickel and iron – under this microscope. A strict magnetic order prevails on these round platelets, which measure from less than 100 nanometres to several micrometres in diameter and only a few dozen nanometres in thickness: “In the plane of the disc, the magnetic moments of the nickel and iron atoms form concentric circles, called vortices,” Matthias Kammerer explains. “At their centre, however, a sharp magnetic needle projects out of the plane of the disc: this is the vortex core.” It is around 20 nanometres in diameter, equating to the diameter of a few dozen atoms.

### Super-speed switching with spin waves

Since the magnetisation of the vortex core can point upwards or downwards, these entities would be ideal for storing data as digital ones and zeros. In principle, a static magnetic field could be used to switch individual points in such memory. “But this field would have to be very strong in or-



A data point changes its polarization: The sample section shows the magnetisation as it reverses from top to bottom.

der to overcome the stability of the aligned peaks,” Kammerer says. This would hamper the processing of such data and would consume a lot of energy.

Yet, there are tricks for overcoming the resilience of these magnetic needles. A number of years ago, the scientists discovered a mechanism that allows the data to be switched using much weaker magnetic fields – at the high speeds that are possible in the fastest present-day magnetic memories. Matthias Kammerer and his research colleagues then found a way to reverse the vortex core directions considerably faster still. They applied a short, rotating pulse of an alternating magnetic field of a few gigahertz frequency to the permalloy discs. “This produced spin waves that amplify in the centre of the disc, similar to the wave patterns in a glass of water if you push it suddenly,” Kammerer explains.

Using these, they managed to switch the magnetic needles in just 0.2 nanoseconds. The researchers have explained the phenomenon theoretically and their calculations suggest they can speed up this switching by yet another order of magnitude. “We have thus laid the basis for developing novel, extremely stable and non-volatile memories that can be switched extremely fast,” says Kammerer. *rb*

Nature Communications, 2: 279, (DOI: 10.1038/ncomms1277): Magnetic vortex core reversal by excitation of spin waves, M. Kammerer et al.

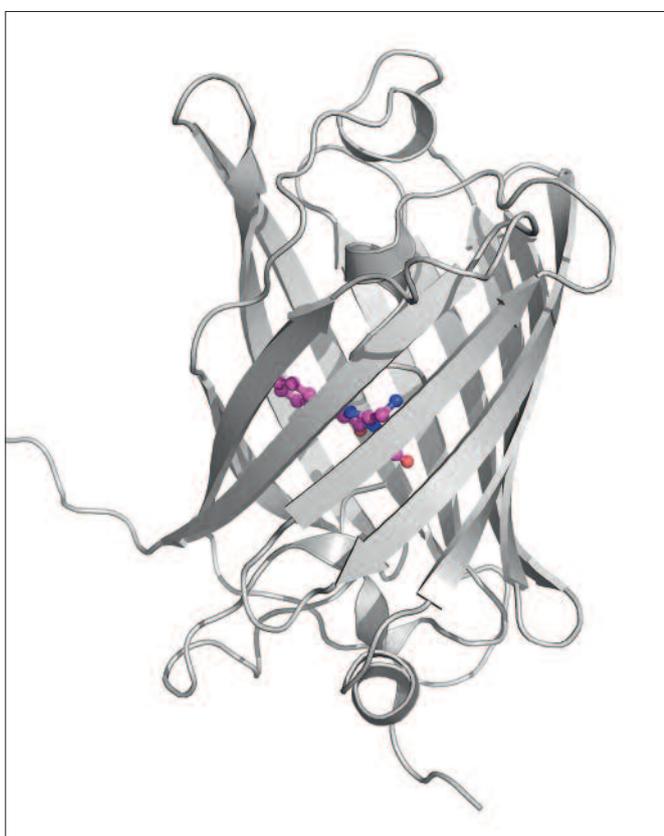
## SUCCESSFUL RESEARCH AT MX BEAMLINES

In the **macromolecular crystallography** lab founded in 2010 at the electron storage ring BESSY II, a research team identified the structure of the fluorescent protein “Dreiklang”. Another group discovered how the protein rhodopsin, which occurs in the human body, absorbs light signals.

**P**roteins are vital components in the human body involved in many biological and chemical processes – they transport oxygen in the blood, trigger reactions in the nervous system as messengers, or fight pathogens as antibodies. Studying proteins is therefore the explicit objective of the Joint Berlin MX Laboratory of the Laboratory for Macromolecular Crystallography, officially inaugurated at the electron storage ring BESSY II in March 2010. The lab is a cooperation between HZB, Freie Universität Berlin, Humboldt University, the Max-Delbrück Centre in Berlin-Buch and the Leibniz-Institut für Molekulare Pharmakologie (FMP) in Berlin-Buch. Three experimental stations at HZB are available specifically for studying macromolecular biological processes, of which the two MX beamlines 14.2 and 14.3 can be used for experiments by researchers of the partner institutes involved. The intense X-ray light from the synchrotron radiation source BESSY II is ideal for researching the spatial configuration of proteins, most of which comprise more than 100 amino acids. Such X-rays can also be used to study proteins in crystal form at the atomic level, for example, to measure these molecules atom by atom. This method is called crystallography. Diffraction of the synchrotron beam at the protein leaves behind a scattering pattern on detectors, from which the protein structure can be determined.

### Structural image obtained of a fluorescent protein

Using crystallography, researchers from the Max Planck Institute for Biophysical Chemistry in Göttingen and of the Freie Universität Berlin studied the structure of the designer protein named “Dreiklang”. This synthesised protein is special for its fluorescent behaviour. Scientists can couple such fluorescent proteins to other proteins to study the processes of life inside cells and organisms at the molecular level. The fluorescent proteins are made to light up at specific target sites or to become dark again where necessary. Basically, they can be switched on and off like a light bulb.



Band model of the fluorescent protein “Dreiklang”, the structure of which was revealed at the electron storage ring BESSY II.

To understand how this effect works, the scientists worked at the MX beamline BL 14.2 to study for the first time the structural characteristics involved in fluorescence on a crystal of the Dreiklang protein when switched on and when switched off. They first switched the green-fluorescing protein crystal from fluorescent to non-fluorescent state at room temperature – they “switched it off”. Next, the scientists measured the crystal when deep-frozen at around minus 170 degrees Celsius on the beamline. “Normally a protein crystal breaks when heated back up to

room temperature after measurement,” Dr. Uwe Müller, head of the HZB “Macromolecular Crystallography” workgroup, describes the special nature of the study: “In this case, however, we managed to keep the protein functional.” They namely brought the protein crystal back into fluorescent state at 30 degrees Celsius, then froze it and studied it a second time on the beamline. The research group’s subsequent data analysis revealed that, when switched on or switched off, the protein’s structure changed by the number of water molecules embedded in it.

“Our study of the Dreiklang molecule broke new ground, since it is a designer protein that does not exist in this form in nature,” says Uwe Müller. “The MX beamline lets us study not only natural proteins but even entirely novel materials. Our work has thus taken us another step forward in HZB’s core research area of functional materials,” Müller concludes.

#### Structure studied during transmission of light signals

On the same beamline, scientists from Charité – Universitätsmedizin Berlin were the first to determine the specific conformation an important information carrier protein in the human body has to assume for it to receive a light signal. Working together with colleagues from Humboldt Universität zu Berlin and from universities in Jeonju/South Korea, London and Toronto, they studied the receptor protein rhodopsin.

Rhodopsin is one of the proteins found in the membranes surrounding all living cells. These proteins connect the cells with signals from the environment such as light, odours and flavours, and with signals from within the organism such as hormones, for example. That means they are involved in almost all physiological processes in the body, and are accordingly associated with most diseases as well. So that a receptor such as rhodopsin can receive information, it must assimilate the signal coded in a molecular information carrier – such as a hormone or a light-sensitive “antenna”. This is only possible if the receptor forms a binding site into which the binding molecule (the ligand) can fit. The research group managed for the first time to hold the light receptor rhodopsin stably in its light-activated state and to explain its structure. In this so-called meta-state, the receptor binds retinal, a derivative of vitamin A, in a form transformed by light.

#### Treatment for pathological changes in signal transfer

With their study, the researchers gained an insight into how the receptor and its ligand interact, and thus into the mechanism by which the signal is transferred into the cell.



Signal transfer: Rhodopsin in Meta II light-activated state. The protein forms a binding site into which a light-transformed version of vitamin A derivative retinal (blue spheres) fits.

“From our example, we can learn how signal transfer from a ligand into a receptor protein can work at all,” explains Prof. Klaus Peter Hofmann, acting director of the Institute of Medical Physics and Biophysics of Charité and member of the Centre for Biophysics and Bioinformatics of Humboldt University. “There are reasons to assume that the fundamental processes of ligand binding are similar for different receptors. Naturally, we also hope to benefit from understanding the fundamental structures and mechanisms for treating pathological changes in signal transfer.” Yet, there is still a long way to go before actual drugs can be developed for treating such pathological changes in signal transfer. *cn*

Nature Biotechnology 29, 942–947 (2011) (doi:10.1038/nbt.1952): A reversibly photoswitchable GFP-like protein with fluorescence excitation decoupled from switching, T. Brakemann, A. C. Stiel, G. Weber, M. Andresen, I. Testa, T. Grotjohann, M. Leutenegger, U. Plessmann, H. Urlaub, C. Eggeling, M. C. Wahl, S. W. Hell & S. Jakobs

Nature, Vol. 471 (7340): 651–52011 (2011) (doi: 10.1038/nature09789): Crystal structure of metarhodopsin II, H.-W. Choe, Y. J. Kim, J. H. Park, T. Morizumi, E. F. Pai, N. Krauß, K. P. Hofmann, P. Scheerer & O. P. Ernst

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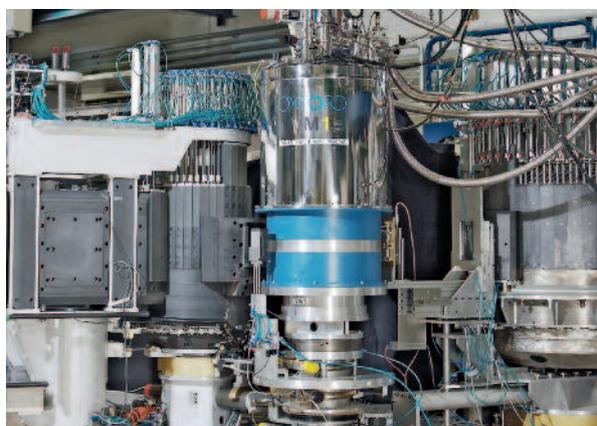
## SPINS IN URANIUM UNDER THE NEUTRON MICROSCOPE

An international research team analysed an unusual uranium compound at HZB. Their results could point the way towards new **high-temperature superconductors**.

**M**ore than 25 years after high-temperature superconductors were first discovered, the mechanisms that cause all electrical resistance to vanish in these materials remain a mystery to this day. Now, we hope that by studying unusual metallic substances whose superconductivity goes hand-in-hand with ferromagnetism, we will be able to tailor superconductive materials to their specific technical applications. As part of this study, a research team working at HZB took a close look at UCoGe – an exotic chemical compound of uranium, cobalt and germanium. “What makes this material special is the fact that superconductivity and ferromagnetism coexist at normal ambient pressure and room temperature,” says Dr. Kirrily Rule, who until recently worked at the HZB department Research with Spallation Neutrons. Indeed, UCoGe actually appears to need this ferromagnetism to become superconducting in the first place. This is quite baffling because, in most other superconducting materials, a strong magnetic field like the kind inside a ferromagnet causes the resistance-free state to collapse.

### Direction dependency detected

To solve the puzzle of why UCoGe is different, Rule and the international team of researchers from the USA, Great Britain and France used HZB’s three-axis spectrometer FLEX. With it, they analysed cold – i.e. relatively slow – neutrons scattered in a sample of this material, to reveal the secrets of this uranium compound and its atomic properties. “The advantages of the FLEX spectrometer are its good signal-to-noise ratio, with few interfering neutrons in the background,” says Dr. Chris Stock of the NIST Center for Neutron Research in Gaithersburg/Maryland (USA), who was involved in the experiments. “This allows us to observe even weak signals” – such as those from UCoGe, for instance, whose atomic lattice scatters so few neutrons that researchers had to resort to a trick to detect them: They arranged two separate crystals of the same material in the apparatus so that they could investigate them as a single, larger crystal. To minimise the risk of environmental neutrons corrupting measurements, FLEX’s



The superconducting compound of uranium, cobalt and germanium was studied on the three-axis spectrometer FLEX.

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detector is shielded by a massive jacket of borated polyethylene.

The effort paid off. “We were able to detect a significant anisotropy, i.e. direction dependency, of the neutron scattering,” reports Rule. From this, the scientists conclude that the magnetic moments of the electrons in the uranium – responsible for its ferromagnetic property – also exhibit strong anisotropy: “This characteristic could be responsible for the tenacity of superconductivity in the material,” adds Stock. And it provides new clues about the fundamental electronic structure; clues that could be of great importance for developing novel superconducting materials. Even the nature of superconductivity in other materials will be better understood, the researchers believe. Accordingly, they will continue to study ever more superconducting materials at the neutron spectrometer at HZB, until their studies yield tailored materials as the final breakthrough in bringing high-temperature superconductors into everyday applications.

*rb*

Phys. Rev. Lett. 107, 187202, 2011 (DOI: 10.1103/PhysRevLett.107.187202): Anisotropic Critical Magnetic Fluctuations in the Ferromagnetic Superconductor UCoGe, C. Stock, D.A. Sokolov, P. Bourges, P.H. Tobash, K. Gofryk, F. Ronning, E.D. Bauer, K.C. Rule, A.D. Huxley

## IMPROVING LUBRICATION WITH ADDITIVES

At HZB, a research team led by Dr. Ali Zorbakhsh of London University has studied the **interface** between oil and metal.

Lubricants help to minimise material wear in machines, save energy in motors, or increase the efficiency of, say, wind turbines. While they have been successfully used for centuries, it is still not fully understood how they work. Ali Zorbakhsh has made it his mission to solve this mystery. The chemist from London University is studying how oil coats surfaces and how the action of oil as a lubricant can be optimised by various additives. These additives influence, for example, the surface tension, longevity and consistency of the oil.

Actually, oil and metal do not like each other, as one may have noticed in the metal salad bowl. Oil draws itself together into droplets instead of evenly wetting the surface, as is necessary for good lubrication. Additives such as palmitic acid help to change that. These long-chain molecules have two ends, one of which likes to dock onto the metal and the other which attracts the oil. Now, Zorbakhsh has studied for the first time how palmitic acid actually behaves on the atomic scale at the oil/metal interface.

He and his British colleagues worked with researchers from Berlin and Belgium as well as partners from industry in the scope of an EU project. They prepared sheets of iron with a thin oxide layer in a special sample holder and dipped them into oils containing palmitic acid added in at different amounts. They studied the interface using neutron reflectometry at Helmholtz-Zentrum Berlin. “Here, we have excellent experimental methods and the experts to go with them,” says Zorbakhsh. The experiments were done on the V6 instrument at the neutron source BER II under the direction of Roland Steitz. “This experiment can only work at all with neutrons, since only they can make it through the metal layer undisturbed to the interface to interact with the molecules there,” Steitz explains. It would be more complicated to study the boundary layer through the liquid without vaporising the temperature-sensitive oil with high-energy beams.

### Good basis for practical applications

“First, we have shown that our method does actually yield information about the oil/metal interface,” says Steitz. The researchers discovered that a boundary layer of palmitic

acid molecules deposits onto the slightly porous iron oxide surface out of the oil phase above it. First, the fatty acid molecules dock onto the iron oxide surface and form a closed, single layer. At this point, the molecules are not perpendicular to the surface, rather tilted. The second layer of palmitic acid above it forms arms that reach loosely into the oil. Generally, the amount of additive adsorbed increases with its concentration in the oil.



Preparing to study additives on the V6 reflectometer.



“These initial findings are a good basis for studying other additives and admixtures typically used,” Zorbakhsh resumes. “We shall now also study in more detail how the additive layer influences the lubricating action.” In particular, the researchers will study in practical experiments at HZB what effects certain stresses such as pressure, shear forces and higher temperatures have on the interface and the lubricating behaviour. The results could then help industry optimise the concentrations of these additives and their compositions for the different lubricating applications. *ud*

Langmuir 2011, 27 (10), 6085-6090: Surfactant Adsorption at the Metal-Oil Interface, M. Campana et.al.

## TWO AGGREGATES IN COEXISTENCE

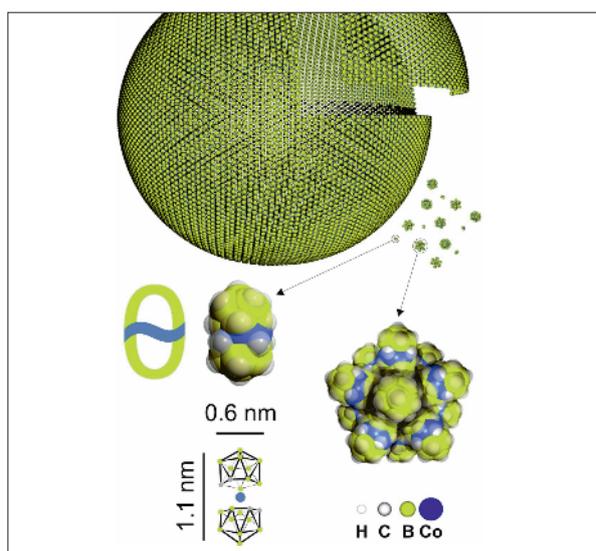
A research team working with Dr. Pierre Bauduin has observed the **unusual aggregation behaviour** of a borane compound at the large facilities of HZB.

**S**elf-assembly is the formation of structures or patterns without external influences and occurs very frequently in nature, especially in the case of molecular surfaces. Micelles and vesicles are some such structures. Vesicles are relatively large, spherical arrangements of surface-active molecules in a liquid, while micelles are small aggregations of amphiphilic, i.e. simultaneously water-soluble and fat-soluble molecules, that also form spontaneously. Micelles, however, only form above a certain material concentration in the solvent. Such processes are of special interest to science and industry since they can be used in technical applications.

While using the large facilities at HZB to study the aggregation behaviour of the cobalt bis(dicarbollide) anion ( $[3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{11})_2]^-$ , COSAN $^-$ ), an international research team led by Dr. Pierre Bauduin of the French Institut de Chimie Séparative de Marcoule (ICSM) discovered the formation of monolayer vesicles and smaller micelles of water-soluble metal carboranes. Carboranes are compounds of boron and hydrogen in which one or more boron atoms have been replaced by carbon atoms. The formation of such structures was hitherto unknown for these compounds. “We chose the cobalt bis(dicarbollide) anion specifically because this commercial substance will soon be finding its way into interesting fields, including pharmaceuticals, synthetic chemistry and elimination of nuclear waste,” says Dr. Sylvain Prevost of the Institute for Soft Matter and Functional Materials at HZB.

### Shape and charge density are crucial

In order to research the aggregation behaviour of the material, called COSAN for short, the scientists dissolved it at varying concentrations in heavy water (deuterium oxide) and studied the samples at HZB using a combination of small-angle and wide-angle X-rays and neutron scattering. “With the X-rays, we can see where there are especially many electrons. In the present case, this was in the cobalt ion. We use neutrons to observe the hydrogen in the borane compound. Employing both techniques gives us a clearer picture of the system,” explains Prevost.



The cobalt bis(dicarbollide) anion is built like a sandwich. It comprises two curved half-cages with a cobalt (III) ion as the “filling” in the middle. At low concentrations in heavy water, relatively large vesicles form, while smaller micelles start to form as the concentration increases.

In doing so, they discovered that, at low concentrations, the partially hydrophobic COSAN first forms vesicles in order to present as little surface to the water as possible. “Only as the concentration rises do the vesicles break down, due to over-density of their negative electric charges, and form smaller micelles. These diminish the interactions of the molecules, but still present a smaller surface than would be the case with individual molecules floating around in the water,” Prevost explains. Within a certain bandwidth of concentrations, both forms therefore coexist. “The unusual aggregation behaviour of COSAN arises from the combination of its particular shape, charge density and form stability.” This makes the material interesting for a series of technical applications – from the recycling of nuclear fuels to ion-selective electrodes, and perhaps even as an HIV protease inhibitor. Further research will have to be conducted, however, to develop practical applications for it in materials research as well. *cn*

Phys. Rev. Lett. 107, 187202, 2011 (DOI: 10.1103/PhysRevLett.107.187202): Anisotropic Critical Magnetic Fluctuations in the Ferromagnetic Superconductor UCoGe, C. Stock et.al.

## INSIDE A DINOSAUR EGG

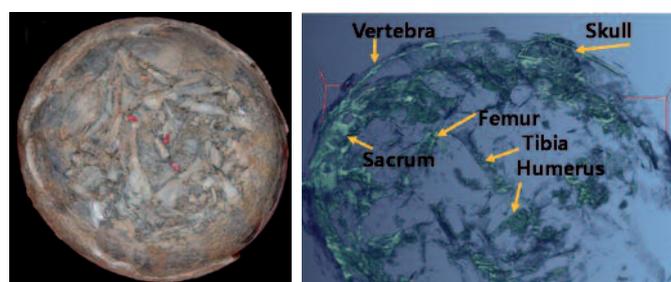
Dr. Nikolay Kardjilov of HZB helped colleagues in South Korea study the fossil of a 100-million-year-old **titanosaurus embryo**.

A dinosaur egg is not a particularly exciting find for a palaeontologist – unless there is still an embryo in it. Such finds are extremely rare. In the present case, the almost spherical egg of nearly 90 millimetre diameter was found in the Gobi desert back in the 1960s. Yet, it had to wait until 2011 before its occupant – the skeletal remains of an unborn member of the titanosauria – could be analysed in South Korea. The egg is part of the collection of the National Science Museum of Korea in Daejeon. That is also the location of the neutron source called the “High-Flux Advanced Neutron Application Reactor” (HANARO) of the Korea Atomic Energy Research Institute, where the fossil was to be studied. The egg consisted of a thin calcite layer with a hollow chamber, on the bottom of which tiny bones were gathered. A typical preparation was not possible, since the remains were too tiny and fragile. The researchers involved therefore opted for the non-destructive method of neutron tomography. They had made no headway using the X-rays of a computer tomograph, since the density of the fossil material was too similar to the surrounding stone. The sample was also too large for studying with synchrotron light.

Neutron tomography, on the other hand, offered the ideal prerequisites, since it can handle samples the size of the dino egg and reveals sharp contrasts between fossilised bone material and the surrounding stone. This is due to the water of crystallisation in the mineralised bones. “The neutrons are highly sensitive in relation to hydrogen,” explains Nikolay Kardjilov of the Institute for Applied Material Research of HZB.

### Know-how transfer to South Korea

Nevertheless, the researchers were faced with a problem: “The Korean colleagues could not resolve the structures of interest finely enough,” says Kardjilov. The test set-up in Daejeon can produce images at 300 microns per pixel resolution. On the other hand, they knew that the instrument at HZB called “Cold Neutron Radiography” or CONRAD for short, could achieve a resolution of 50 microns per pixel. Unfortunately, for insurance reasons, the museum in Dae-



View of the fossilized titanosaurus embryo – the red arrows in the left picture point out the embryo bones. On the right is a tomograph.

jeon did not feel comfortable having the valuable exhibit shipped to Berlin for study. So, instead, Nikolay Kardjilov went to Korea – with a thin scintillator screen and a high-resolution objective in his luggage. “With our expertise from the CONRAD detector, we were able to help the colleagues substantially,” explains the Berlin researcher.

### 600 individual high-resolution images

In neutron tomography, the sample is studied using a parallel beam of thermal neutrons. During the process, it is revolved little by little through 180 degrees on a turntable. In this case, the dino egg was turned in 600 steps and bombarded with a 25-meV neutron beam. The individual images were then put together on the computer. “At this high resolution, there are a number of challenges,” says Kardjilov. The parallel neutron beam fans out slightly on its path to the sample. At lower resolutions, this effect can be neglected. For the dino egg, however, the image had to be corrected using a cone beam algorithm. This work was done at HZB, where the researchers have plenty of experience in exactly this procedure. The results are some impressive pictures of the egg. “It was like removing a frosted glass plate from the pictures,” Kardjilov rejoices. cs

Gondwana Research 20 (2011), p. 621-629: Description of the first lithostrotian titanosaur embryo in ovo with Neutron characterisation..., G. Grellet-Tinner et.al.



# HIGHLIGHTS FROM OUR OWN RESEARCH

**R**esearchers at Helmholtz-Zentrum Berlin work constantly to keep things running smoothly at the large-scale research facilities. This means not only supervising their operation but also making improvements. The neutron source BER II, for example, was being renovated for all of 2011. The colleagues at HZB boosted the performance of its beamline and neutron guides and improved the research equipment at its measuring stations.

By conducting their own in-house research, the scientists at Helmholtz-Zentrum Berlin are continually expanding their repertoire of methods at HZB in order to maintain the basis for new discoveries. For instance, until recently there had been no means by which to observe the motions of molecules in real time at the crucial moment of a chemical reaction, seeing as they take place in the tiniest fraction of a nanosecond. To provide the means that will crack the mysteries of such little-understood processes in future, a group of researchers at HZB developed a method

for filming molecules using light flashes at femtosecond intervals.

The size and electronic properties of nanoparticles can be tweaked to make them perform essential tasks in solar cells or in the production of hydrogen. Another team of HZB-scientists has used the brilliant X-rays at the synchrotron source BESSY II to improve the spatial resolution of X-ray spectroscopy and thereby improve the precision of future nanoparticle studies.

Fast data storage is becoming ever more important in the information age. When studying barium titanate, HZB researchers discovered an unusual and extremely rare property: The multiferroic crystal exhibited ferroelectric and ferromagnetic properties even at room temperature, making it an ideal building material for data storage. Until then, multiferroic properties had only been observed at very low temperatures.

Keep reading to learn more about these and many other results from the pure research at HZB.

## FASTEST MOVIE IN THE NANOWORLD

HZB researchers have demonstrated how to film **ultra-fast processes** in the realm of molecules and nanostructures using X-ray laser flashes and holographic projection.

It's not often that scientific results make it into the Guinness Book of Records. Physicist Prof. Stefan Eisebitt, head of the HZB research group Functional Nanomaterials and of the work group Nanometer Optics and X-Ray Scattering at TU Berlin, and his team have earned this honour. The Berlin researchers are listed in the 2012 issue of the "bible of world records" as the creators of the "world's fastest movie" for a sequence of images taken only 50 femtoseconds (quadrillionths of a second) apart. The researchers had set out to prove that extremely rapid processes in the nanocosmos can be observed time-resolved using X-ray light flashes. "Many physical, chemical and biological processes that are important for technical applications or for researching and treating diseases take place on the scale of atoms, molecules or nanostructures," says Eisebitt – like complex chemical reactions or protein folding, for example. While methods such as short-wave X-ray diffraction can take still images of these particles only a few millionths of a millimetre in size, there had never been

a way to watch them "live" as they move, collide or change their shape.

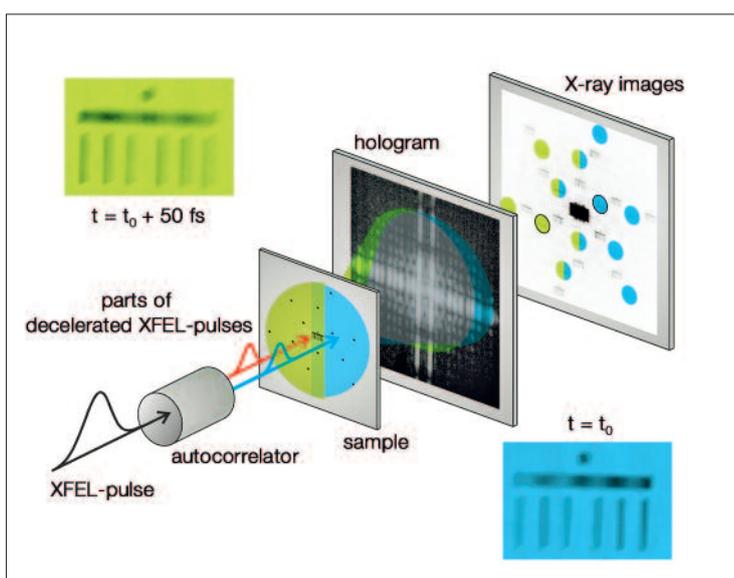
### Pictures taken femtoseconds apart

The researchers working with Stefan Eisebitt found a way to change that. They flashed short pulses of X-ray light onto a miniaturised depiction of the Brandenburg Gate etched into metal foil only microns thick. The light flashes of 23.4 nanometre wavelength and 20 femtosecond duration were produced in the research facility FLASH of the Deutsche Elektronen-Synchrotron (DESY) in Hamburg. Here, electrons accelerated to nearly light speed and periodically wiggled by magnetic fields emit the coveted short-wave laser flashes.

X-ray laser pulses lit the etching multiple times in rapid succession – similar to making a film. "It was actually a problem that the laser flashes used for imaging came at an unimaginably short time interval of a few femtoseconds – much too close together to simply take a sequence of individual pictures with them, as in a camera," says Eisebitt. Even when using a digital camera, it always takes time between two photos to read out the microchip and save the preceding photo. No more could one hope to process the individual frames of a nanoscale video within this time-frame. The processes are simply too fast for any image sensor.

### Mathematically reconstructed pictures

Stefan Eisebitt and his team therefore used a trick: The Berlin scientists produced two pictures of the miniature Brandenburg Gate, but they did not save them sequentially as two separate pictures, rather they superimposed them into a single hologram. To do so, the researchers used a mirror to split the X-ray beam into two partial beams. Other mirrors diverted one of the two beams onto a separate path to make it reach the object 50 femtoseconds later than the other.



Experimental set-up: The X-ray laser pulses first strike the sample at 50-femtosecond intervals and then overlap into a hologram from which the images are calculated.

The hologram was produced in a light-sensitive chip behind the object, which the two partial beams struck after lighting the object. Using mathematical operations, the two pictures of the Brandenburg Gate were reconstructed from the spatial pattern of the two holographically mixed instantaneous snapshots.

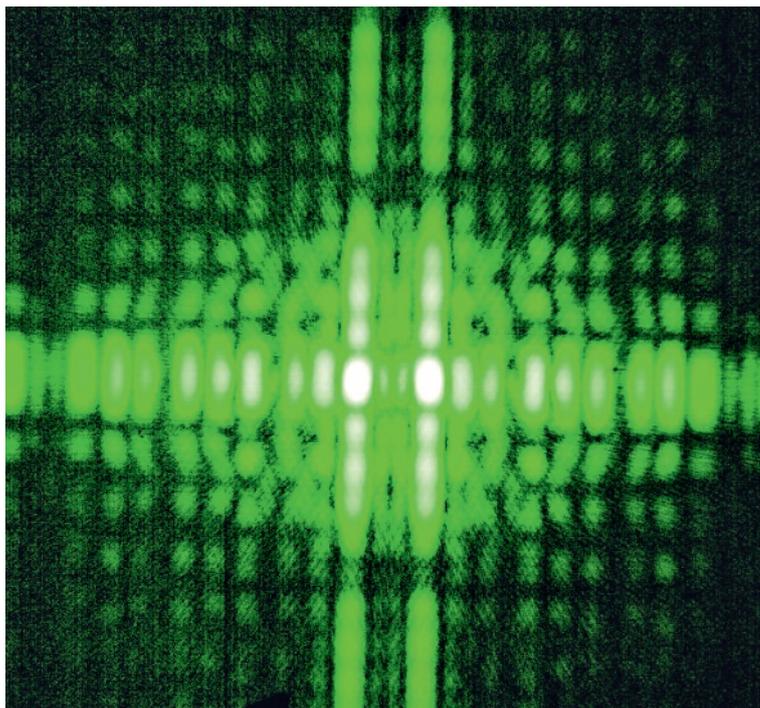
The “movie” thus produced is absurdly short and kind of boring, admits Stefan Eisebitt with a smirk. It only shows two pictures of the exact same thing – the model they “filmed” did not even change between the two pictures. It is also much larger than a molecule or a collection of atoms. “But what we primarily wanted to show with our experiment was that the method works for producing movies of nanometre-sized structures at extremely high temporal resolution,” stresses the Berliner physicist. And indeed his team did so with flying colours.

Stefan Eisebitt and his team are hard at work enhancing the experimental method. They have also turned their attention to explaining magnetic phenomena such as the ultra-fast demagnetisation of certain ferromagnetic materials. Physicists still do not fully understand this effect, discovered in the mid 1990s. “By this remarkable process, the magnetisation in thin magnetic layers disappears in a few dozen femtoseconds,” explains Eisebitt. Understanding what causes this effect would bring the researchers closer to answering what is the physical speed limit on a change in magnetisation, such as switching a data-bit in memory, for example.

### Magnetic memory under scrutiny

Dutch researchers led by Dr. Ilie Radu of the work group Methods and Instrumentation of Synchrotron Radiation at HZB took a close look at ultra-fast magnetic processes. They used the electron synchrotron BESSY II in Berlin – and experimental methods similar to those of Stefan Eisebitt. While analysing the physical processes that take place in magnetic memory media, Radu’s team encountered a hitherto unknown phenomenon.

In these kinds of memory, data is encoded by a certain alignment of atomic magnetic moments. The moments of neighbouring atoms are aligned either parallel or antiparallel, corresponding to a digital “1” or “0”. This alignment is governed by a strong quantum mechanical force called the exchange interaction. The researchers wanted to know



Captured hologram, from which the scientists reconstructed two images of the Brandenburg Gate model taken at a 50-femtosecond interval.

what happens when they overcome this force. They fired short laser pulses of only a few femtoseconds duration at an alloy of gadolinium, iron and cobalt. These heated the metallic material, whereupon the magnetisation changed. The initial magnetisation of the sample was based on an antiparallel alignment of the gadolinium and iron atoms. After lasering, the scientists watched the change in the material using ultra-short X-ray flashes. They discovered that the magnetic moments changed their direction at different speeds. The iron took less than 300 femtoseconds to change, while the gadolinium atoms took five times longer. Thus, using ultra-short laser pulses as an external trigger, they managed to reverse the magnetisation so quickly that it took place on a different time scale for each of the two components in the ferromagnet. “This is as strange as finding the north pole of a magnet reversing slower than the south pole,” says Radu. “This finding points the way towards developing novel computer memories that can process data about a thousand times faster than they can today.” Do we have a glimpse at another Guinness world record? rb

Nature Photonics 5, 99–102, 2011 (doi:10.1038/nphoton.2010.287): Sequential femtosecond X-ray imaging, C. M. Günther, B. Pfau, R. Mitzner, B. Siemer, S. Roling, H. Zacharias, O. Kutz, I. Rudolph, D. Schöndelmaier, R. Treusch & S. Eisebitt

Nature 472, 205–208, 2011 (doi: 10.1038/nature09901): Transient ferromagnetic-like state mediating ultrafast reversal of antiferromagnetically coupled spins, I. Radu, K. Vahaplar, C. Stamm, T. Kachel, N. Pontius, H. A. Dürr, T. A. Ostler, J. Barker, R. F. L. Evans, R. W. Chantrell, A. Tsukamoto, A. Itoh, A. Kirilyuk, Th. Rasing & A. V. Kimel

## MULTITALENTS FOR DATA MEMORIES

Materials that are both ferromagnetic and ferroelectric are excellent for building faster and lower-energy data memories. HZB researchers have laid the foundation for this by demonstrating “**multiferroic**” material behaviour at room temperature.

**M**ultiferroic materials are considered wonder materials for microelectronics, since they unify two physical properties useful for processing digital data: ferromagnetism and ferroelectricity. While a ferromagnetic material can be magnetised by an external magnetic field, such as iron under the action of a rod magnet, a ferroelectric crystal spontaneously changes its polarisation when placed in an electric field; a partial separation of positive and negative electric charges occurs. Only very few substances have been found to exhibit both ferromagnetism and ferroelectricity, since the two phenomena require different conditions. If they ever do occur together, then mostly only at very low temperatures.

### Multiferroic at room temperature

Scientists at HZB working with physicist Dr. Sergio Valencia Molina have demonstrated such multiferroic properties at normal room temperature. They did this at the electron storage ring BESSY II, where they scattered X-rays on barium titanate ( $\text{BaTiO}_3$ ). The researchers employed “soft X-ray resonant magnetic scattering” to study the magnetic moment of the titanium and oxygen atoms in a film of barium titanate. This crystalline material is in fact a known ferroelectric. To give it ferromagnetic properties as well, the scientists used a trick: They applied a nanometre-thin layer of iron or cobalt – both ferromagnetic metals – onto a thin film of barium titanate. “We will need such thin layers for future technological applications in electronic devices as well,” Valencia says.

“We have shown for the first time that certain regions of barium titanate can have multiferroic properties,” the physicist is pleased to announce. In this particular case, the effect occurred within a transition layer to the iron or cobalt in the film. This layer is only a few atomic diameters thick. In it, the magnetic properties of the material could be controlled by an electric field – which is much easier than controlling them by magnetic fields, as has so far been the case for reading or writing conventional memory media. When an electric potential is applied to the barium titanate film to reverse its electric polarisation, its magnetisation



HZB scientist Florin Radu inspects a  $\text{BaTiO}_3$  sample in the ALICE diffractometer.

changes as well. And, as the experiments have proven, this even works at room temperature.

### A useful effect

“This effect can be used to store data quickly and with low power,” says Valencia. With a strong coupling between ferromagnetic and ferroelectric properties, the magnetic behaviour can be controlled electrically – and the power requirements could drop by as much as 99 percent. A weak coupling, however, would allow an entirely novel concept in data storage – using four different states of the data bit instead of the usual two. The HZB researchers still do not know exactly how strong this coupling is. Nevertheless, the discovery provides a novel way to create multiferroic substances at room temperature – and makes barium titanate highly interesting as a material for memory media. *rb*

Nature Materials 10, 753–758, 2011 (doi:10.1038/nmat3098): Interface-induced room-temperature multiferroicity in  $\text{BaTiO}_3$ , S. Valencia et al.

## LASER BEAM MAKES CELLS “BREATHE IN” WATER

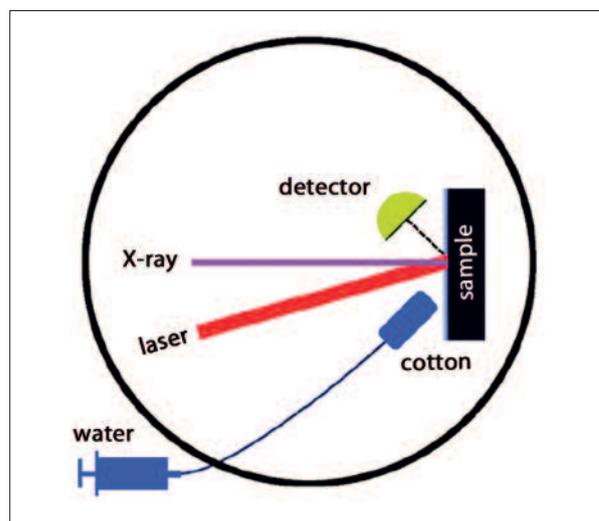
Scientists have shown how laser light influences **interfacial water** using experiments at BESSY II. Their results could help facilitate the uptake of cancer drugs.

**W**ater is the basis of all life, yet not all water is equal. Scientists make a distinction between free, liquid water (bulk water) and the extremely thin layers of water molecules that adhere to boundary surfaces of other materials. This so-called interfacial water is of great interest since it makes up a large portion of the water in biological systems such as cells, for example. It resides on the cell membrane and on other molecular structures inside the cell. Scientists have already demonstrated in various experiments that certain properties of interfacial water, such as viscosity or heat capacity, differ from bulk water. These properties also depend on the material and structure of the respective boundary surface.

### A laser beam as disruptive pulse

Studying interfacial water is a major challenge for scientists. Being only a few atoms thick, these water layers are notoriously difficult to control in experiments. A research team led by Dr. Andrei Sommer of the Institute of Micro and Nanomaterials of the University of Ulm worked together with Prof. Dr. Emad Flear Aziz Bekhit and his junior research group Functional Materials in Liquids of HZB to study interfacial water using X-rays.

The researchers used an artificially produced diamond layer as the boundary surface in their experiment. They placed this sample in the Liquidrom – an experimental chamber set up at HZB specifically for liquids – to expose it to a gaseous atmosphere of controlled humidity so that an extremely thin layer of water would deposit onto it. “The diamond surface was prepared to make it hydrophobic and essentially inert to other substances. The water deposited onto it without binding to it,” explains Dr. Kai Hodeck of the junior research group Functional Materials in Liquids. They examined the interfacial water deposited onto the sample with soft X-rays and a laser. “Soft X-rays are especially suitable for studying water. What we measured is the fluorescent light resulting from the irradiation with X-rays. This contains information about the electronic structure of the molecules. At the same time, we selectively disrupted the interfacial layer using a laser in order to observe the



A moistened piece of cotton (blue) ensured a thin layer of water always remained on the sample (black). This was modified using laser pulses (red) and studied by X-rays (violet).

change in fluorescence and thus the structure of the water layer,” explains Hodeck.

### Implications for medicine

While the experiment was performed on a highly simplified model system, the result is noteworthy: Sommer was able to show that the interfacial water expands and becomes “more fluid” upon laser irradiation. Then, when the laser beam is switched off, the water retracts again and becomes “more solid”. This effect could be used, for example, to make the interfacial water in cancer cells ‘inhale’ anti-cancer drugs from the cells’ environment under laser light. “Many further tests will have to be made, however, before we can actually go down this theoretical path. And HZB has the instruments for studying interfacial water by complementary methods such as infrared spectroscopy or neutron scattering,” says Hodeck. *cn*

J. Phys. Chem. Lett., 2011, 2 (6), pp 562–565 (doi: 10.1021/jz2001503): Breathing Volume into Interfacial Water with Laser Light, A. P. Sommer, K. F. Hodeck, D. Zhu, A. Kothe, K. M. Lange, H.-J. Fecht & E. F. Aziz

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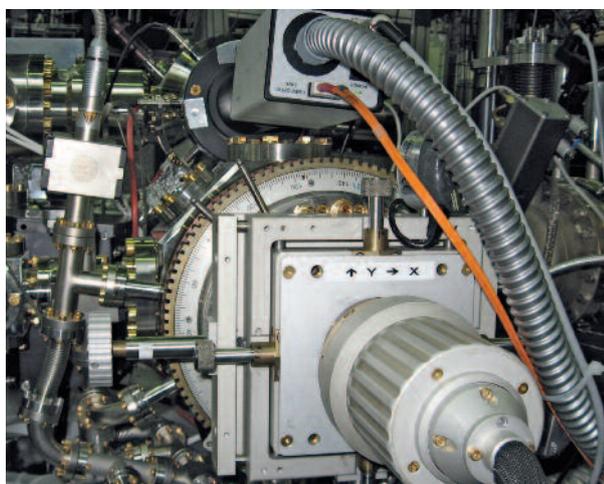
## TWO METHODS ARE BETTER THAN ONE

A team led by **Dr. Florian Kronast** has developed a microscopy method at HZB for studying the magnetic properties of individual iron nanocubes.

Whether in cancer therapy, as sensors or for data storage, magnetic nanoparticles serve a wide range of applications. To optimise them for any given application, one has to know their exact geometrical structure as well as their electronic and magnetic properties. Yet, nanoparticles have always been tricky things to characterise individually. They are simply too small for a lot of measuring techniques. Instead, experimenters tend to measure several thousands of particles at a time before making calculated conclusions on their individual properties. “But that is not enough to optimise nanoparticles to their specific applications, or to study their magnetic properties, structures and interactions between them,” says Florian Kronast of the Department for Magnetisation Dynamics at HZB. “The properties depend not only on the particle itself, but also on how it is positioned on a surface and how it relates to neighbouring particles. The particles can influence one another considerably.”

### Magnetic sample holder developed

Kronast and his colleagues have therefore developed a new microscopy method for characterising individual particles.



X-ray photoemission electron microscopy (X-PEEM) at HZB.

Such as the iron nanocubes produced and studied by the team of Michael Farle of the University of Duisburg-Essen, for instance. The particles in solution are rinsed onto a sample and then measured simultaneously by the hundreds. Their individual properties can then be determined from the surface images. To correlate the size, shape and alignment of individual particles with their electronic and magnetic properties, the researchers combine two methods. Using scanning electron microscopy (SEM), they obtain a high-resolution image of the sample surface and thus information about the size, shape and distribution of the particles. X-ray photoemission electron microscopy (X-PEEM) serves to study the electronic and magnetic properties of the specific elements.

The sample is also subject to a magnetic field that is varied in strength and direction, in order to record the magnetic reaction of the particles. The twist: “Using a newly developed sample holder, we can apply a local magnetic field that covers only a few hundred microns and which does not distort the results by disrupting the escaping electrons,” says Kronast.

The novel measurements showed that the iron cubes of 18 nanometre edge length exhibit preferred magnetic directions, and that the stability of their magnetisation at room temperature depends greatly on interactions with neighbouring particles. “This shows how important it is to be able to study the particles individually and not only as averages over a larger ensemble,” Kronast continues. Only then can their interactions be analysed as well. “Our experiment offers unique access to studying magnetic nanoparticles and their magnetic interactions,” Kronast concludes. X-PEEM is installed as a permanent experiment at BESSY II and the special magnetic sample holder is in high demand among the user community.

*ud*

Nano Letters, 2011, 11 (4), 1710–1715 (DOI: 10.1021/ nl200242c): Element-Specific Magnetic Hysteresis of Individual 18 nm Fe Nanocubes, Florian Kronast et.al.

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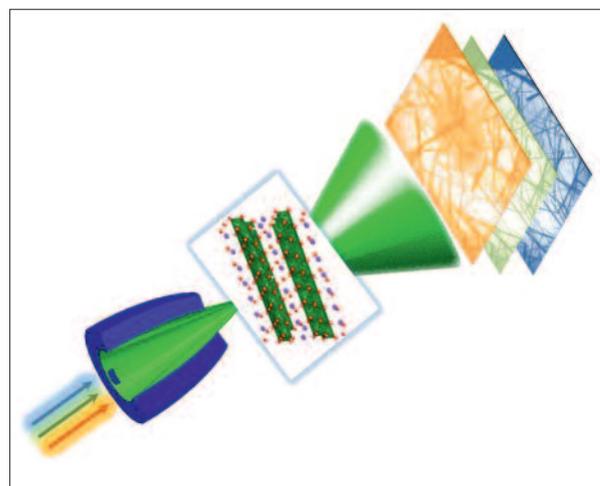
## SHARP IMAGES OF THE TINIEST OBJECTS

At HZB, **Dr. Peter Guttman** and the microscopy team of Dr. Gerd Schneider have made an important breakthrough in X-ray nanospectroscopy.

**N**anoparticles are coveted helpers when it comes to increasing the efficiency of solar cells, lithium-ion batteries or hydrogen production processes. What makes them so attractive is their extremely small size. Relative to their volume, they have a very large surface area for chemical reactions to take place on. This makes them more efficient as catalysts. The smaller the particles, however, the more difficult they are to study in detail. Gaining insights into the geometric and electronic properties of nanoparticles is still a challenge for researchers. But we need these insights if we are to tailor them to specific applications. Dr. Peter Guttman and his colleagues at Helmholtz-Zentrum Berlin have now developed a novel microscopy method by which they can measure many nanoparticles together and yet still individually. It combines the high spatial resolution of X-ray microscopy with NEXAFS (near-edge X-ray absorption fine structure) absorption spectroscopy, and uses the brilliant X-rays from the synchrotron source BESSY II to illuminate the samples. What they do is change the photon energy of the X-rays in tiny steps. A high-resolution objective projects an image of the irradiated sample onto an X-ray camera. For this, the researchers have been using zone plates manufactured using a specially made, high-precision electron beam lithography system at HZB. Now, they are re-developing the objectives to improve the spatial resolution from the present 25 nanometres down to 10.

### Faster image build-up

“What is special about our new microscope is its capillary optic through which the already monochromatised X-ray light is shone onto the sample,” explains Guttman. It was developed together with a firm in the U.S. “Its advantage over the zone plate condensers used so far is that it always focuses the X-ray light equally well on the sample, irrespective of its energy,” Guttman stresses. The novel microscope can image sample areas of 20 by 20 microns at a time, covering very many nanoparticles simultaneously, and can run through the appropriate X-ray energy spectrum very quickly. Every single particle can be subsequently analysed from the data obtained. Thus, the researchers ob-



Schematic diagram of titanium dioxide rods illuminated by X-rays of various photon energies through a capillary condenser. A high-resolution lens – not shown here – then projects an image of the objects.

tain both individual and statistically significant information about the sizes and electronic properties of the nanoparticles and their distribution. “Compared to scanning X-ray microscopy, where the image is built up sequentially pixel by pixel and is thus extremely time-consuming, we are a hundred times faster while also having better spectral resolution,” Guttman continues.

The Helmholtz researchers have already demonstrated the performance of the new technique in various projects. Together with colleagues from Belgium, France and Slovenia in the scope of the European cooperative COST action NanoTP, for example, they studied specially shaped nanorods of titanium dioxide. These are to be used in the semiconductor industry to shrink electronic components further than ever. “Since X-ray microscopy is equally suitable for illuminating cells, it also provides the ideal conditions for studying the environmental hazard of nanoparticles,” Guttman believes. ud

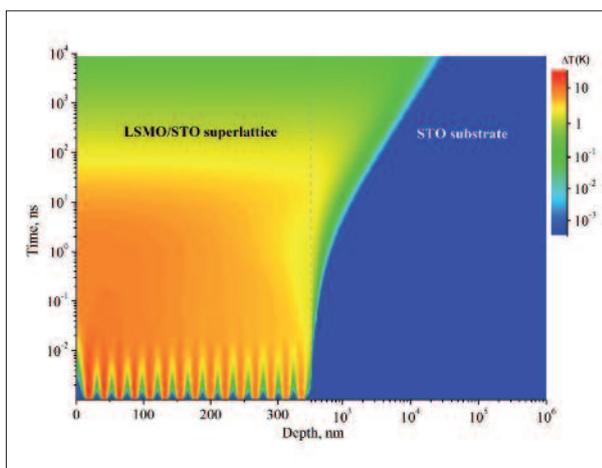
Nature Photonics 6, 25–29, 2012 (DOI: 10.1038/nphoton.2011.268): Nanoscale spectroscopy with polarised X-rays by NEXAFS-TXM, P. Guttman et al.

## HEAT MOVING AT LIGHTNING SPEED

In layers of a ferromagnetic material only nanometres thick, heat disperses considerably faster than in a larger volume of the same material of the same **thermal conductivity**. Researchers demonstrated this significant insight for nanoelectronics at BESSY II.

While the name of the nanostructure may be a bit of a tongue-twister, physicists absolutely revere the compound  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3/\text{SrTiO}_3$ . Layers of it just a few nanometres thick are regarded as hot candidates for producing higher performance components for future nanoelectronics – electronic elements far smaller and therefore faster than today’s conventional processors and memory chips. The coveted material combination, abbreviated to LSMO/STO, is so well suited to these components because LSMO is ferromagnetic and metallic while STO is an excellent insulator.

If this material is to be optimised for specific applications, then its characteristics have to be precisely known first. There is a physical limit on size, for example, below which components made from this material drastically change their electrical behaviour, because quantum effects dominate or because the current heats up the thin layers too much. A group of researchers led by Prof. Matias Bargheer, professor of physics at HZB and at the Institute of Physics and Astronomy of Potsdam University, studied exactly where this limit exists for LSMO/STO at the electron storage ring BESSY II. The researchers demonstrated that, even



The contour diagram shows the calculated temperature increase of the studied LSMO/STO sample relative to time and depth of the material.

when stacked into layers of around 10 nanometres thickness, this wonder material exhibits no strange properties – at least in terms of heat transfer.

### Watching heat distribution

The researchers shone infrared laser light onto a nanostructure applied onto a substrate as a so-called superlattice of 15 alternately overlapping layers of LSMO and STO. The light struck the substance in short, rapid flashes, warming it by about six degrees Celsius for a billionth of a second. At a slight delay after each flash, they shone ultra-short pulses of high-energy X-rays from BESSY II onto the sample in precisely coordinated sequence with the laser flashes. A detector caught the X-rays that were diffracted at the atoms of the LSMO/STO crystal lattice. This allowed them to measure changes in the diffraction pattern at high temporal resolution. These changes resulted from variations in the mean separation of the lattice atoms, which depend sensitively on temperature. “In this way, we achieved an extremely high-precision temperature measurement within a hundredth of a degree Celsius,” says Bargheer – and that was time resolved to a scale between 100 picoseconds and 4 microseconds. The researchers could therefore watch how the heat distributed itself throughout the superlattice and how it penetrated into the substrate.

“The results of the measurements show that the heat dissipates very rapidly,” reports Bargheer. The temperatures of the two materials in the nanolayer had equalised within 100 picoseconds (trillionths of a second). “That agrees excellently with simulations of heat transfer in a large-volume sample of the substance,” says the physicist. “That means the mechanism of heat conduction in the nanolayer does not differ significantly from the heat diffusion in the macroscopic material” – a good thing to know if we want to manufacture future nanoelectronic storage elements from  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3/\text{SrTiO}_3$ .

rb

New Journal of Physics 13 (2011) 093032 (doi:10.1088/1367-2630/13/9/093032): Nanoscale heat transport studied by high-resolution time-resolved X-ray diffraction, R. Shayduk et al.

## GOLD PARTICLES IN EGGSHELL

Junior researcher Dr. Yan Lu at HZB has developed a **thermosensitive microgel** that can be used to control the catalytic activity of nanoparticles.

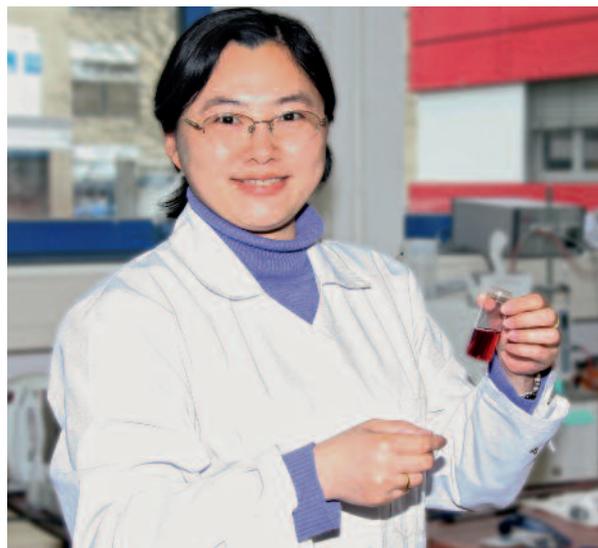
One takes nanoparticles of gold, coats them with a layer of silicon dioxide, and lets a shell of PNIPA (poly(N-isopropyl-acrylamide)) grow on top of them. Next, one etches away the silicon-containing intermediate layer. What remains, the researchers call a yolk-shell structure. The yolk is the nanoparticle, the shell is a dense network of polymers. While this means the shell is not as robust as that of an egg, it is not actually supposed to be. Quite the opposite: It should allow other molecules to get through to the nanoparticle so that it can fulfil its purpose as a catalyst. So that the particle's surface remains free for this purpose – to continue the metaphor – there is no egg white.

All this effort is worth it, since it solves a problem that metal nanoparticles have in practice: They tend to clump together of their own accord. The shell prevents them from doing so. Yet it can do even more: If it is built out of a suitable polymer network, then its permeability can be controlled by external influences, such as its temperature, for example. That way, the catalytic activity of the nanoparticles can also be influenced indirectly.

Junior scientist Yan Lu and her colleagues at the Institute for Soft Matter and Functional Materials of Professor Matthias Ballauff at HZB have produced such organic-inorganic hybrid nanoparticles with a yolk-shell structure. In order to study the influence of the shell on their catalytic activity, Lu observed as a model reaction the reduction of 4-nitrophenol (4-NP) and nitrobenzene (NB) by sodium borohydride (NaBH<sub>4</sub>) to 4-aminophenol (4-AP) and aminobenzene (AB). Both starting molecules have a similar structure, but 4-NP is a hydrophilic molecule, i.e. it “likes” water, while NB has a hydrophobic character.

### Temperature determines permeability

The nitro compounds were each added with NaBH<sub>4</sub> and the nanoparticles into an aqueous solution, the temperature of which was varied. Using UV spectroscopy, the researchers observed how efficiently the reduction reaction occurred. Lu and her colleagues discovered that the reduction of 4-NP is favoured at low temperatures while the reduction of NB oc-



Junior scientist Dr. Yan Lu studied how the catalytic behaviour of gold nanoparticles can be influenced in a targeted manner.

curs at higher temperatures – behaviour that is not observed for pure gold nanoparticles.

How can this be explained? While the network of the PNIPA shell does become denser as the temperature rises, this is not cause enough on its own, since the molecules are approximately equal in size. “What is decisive is that PNIPA changes from a hydrophilic to a more hydrophobic state at the so-called lower critical solution temperature,” explains Lu. When PNIPA is hydrophilic, it allows other hydrophilic molecules such as 4-NP to pass more easily, but when it is hydrophobic, it starts to block these out and to let hydrophobic NB through by preference.

This effect is boosted even further if both molecules are present together in the same solution. “The reaction selectivity of a molecular mixture can thus be very well controlled using a single catalyst,” Lu resumes. “Yolk-shell structures thus offer many possibilities for adjusting the catalytic activity of metallic nanoparticles to specific applications.”

*ud*

Angew. Chem. Int. Ed., 51: 2229–2233, (DOI: 10.1002/anie.201106515): Thermosensitive Au-PNIPA Yolk-Shell Nanoparticles with Tunable Selectivity for Catalysis, S. Wu, J. Dzubiella, J. Kaiser, M. Drechsler, X. Guo, M. Ballauff & Y. Lu

## SOLAR CELLS IN FOCUS AT THE LARGE FACILITIES

How copper atoms migrate through layers or crystal defects influence the **efficiency of solar cells** – HZB researchers have discovered the causes of these processes of great importance to the solar industry.

The equipment set up at Helmholtz-Zentrum Berlin for materials research is probably unique in the world. Many instruments are available to scientists for all kinds of experiments. Pure research into photovoltaics is one example. Researchers are developing new materials for efficient solar cells, or creating simple and cost-effective production methods. This is the aim of Dr. Roland Mainz, scientist at the Institute of Technology, who intends to boost the efficiency of the manufacturing process for the absorber layer in thin-film solar cells. This light-sensitive layer of a few microns thickness serves to convert the energy of sunlight into electrical current.

### Efficient manufacture at a glance

“To further improve the efficiency of such thin films and the reliability of the manufacturing process, it is important to know exactly what reactions are taking place here,” says Mainz. The physicist is focussing mainly on so-called



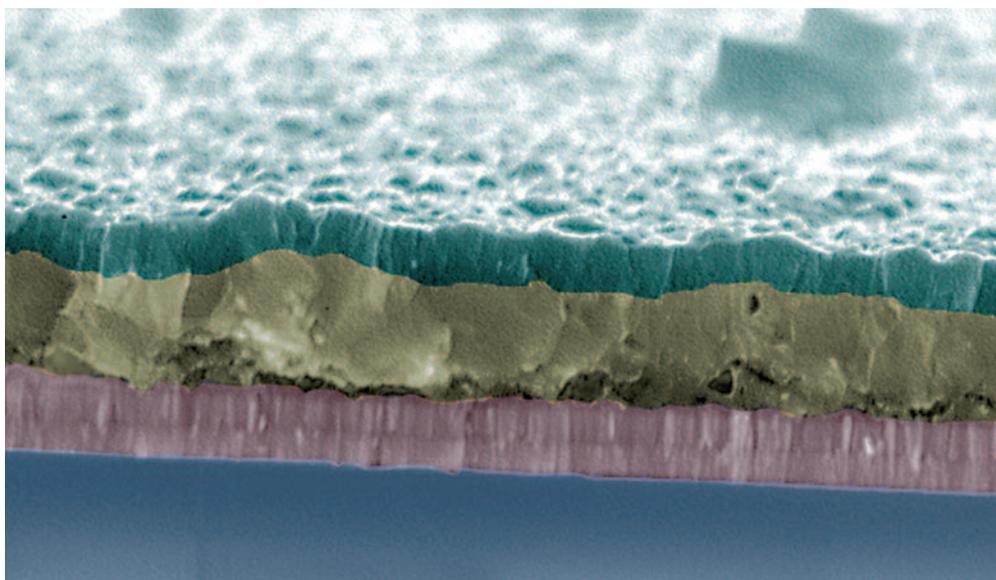
The processes taking place inside solar cells are studied at HZB.

chalcopyrites as absorber materials – compound semiconductors of copper, indium, gallium, and sulphur or selenium that can achieve especially high degrees of efficiency. To produce cells out of these, the substances are usually deposited from gaseous phase onto a substrate using an elaborate process. An alternative, cheaper method is to apply copper, indium and gallium first as individual layers and then heat the resulting material stack inside an atmosphere of sulphur or selenium. The atoms of these substances then diffuse between the layers, which change due to “phase transformations” until the desired absorber layer develops.

“It was long unknown how the distribution of the various chemical elements changes over time in these processes,” Roland Mainz says. There was namely no method by which to observe them directly. Yet the physicist has now overcome this barrier. He has found a way to watch the changes of elemental distribution “live” inside the layers. This is where the X-ray light from HZB’s electron storage ring BESSY II comes in, the energy of which can be varied seamlessly. The Berlin researcher’s trick was to combine X-ray diffraction measurements with fluorescence measurements in the material film, which he radiated with X-rays of varying energy for this purpose.

### Tracking down point defects

Mainz developed a method for reconstructing the onset of various material phases as well as changes in the distribution of chemical elements from the results of these combined measurements. Thus, he was able to illuminate certain details in the formation of the light-absorbing layer, the knowledge of which serves as a starting-point for improving thin-film solar cells. Excess copper atoms, for example, quickly diffuse through the other layers to react ultimately with sulphur or selenium on their surface. “This diffusion of copper appears to improve the characteristics of the layer,” says Roland Mainz. However, undesirable cavities can form in the layer during this process. The aim is to adjust the manufacturing process to put these insights to optimal use. Prof. Susan Schorr, head of the Crystallography Department,



The layer structures of a CIS solar cell – imaged under the scanning electron microscope. When studying the structural features of this compound by neutron scattering, the research group of Prof. Susan Schorr discovered that so-called point defects hardly impair the efficiency of a solar cell.

is also concentrating on chalcopyrites for producing thin-film solar cells. “We are mainly concerned with the structural features of these compounds,” says Schorr – in particular point defects. These are tiny imperfections in the crystalline composition of the material, caused by missing atoms or impurities, for example. “These have a major influence on the properties and efficiency of a solar cell,” the crystallographer says. Yet, no detailed analyses of the concentration of point defects in chalcopyrites had ever been made. “We have now investigated these defects in a systematic study for the first time,” Schorr reports.

The extensive equipment at HZB helped in this endeavour. The major role was played by HZB’s second ultramodern scientific large facility, which complements the synchrotron source: its neutron source. Susan Schorr employed neutron diffraction on crystalline samples. “Only this method allows us to determine the type and distribution of point defects,” she says. Powder samples are produced in the lab for the neutron experiments on cuprate compound semiconductors. This has the advantage that the chemical composition of a sample can be adjusted very precisely.

#### Surprises lurking in the electron bands

The neutron scattering results revealed that a high concentration of copper imperfections existed in the studied material – places in the crystal lattice where a copper atom ought to be, but was in fact missing. “It seems miraculous that solar cells can function so well despite such a faulty material,” Schorr marvels. “Yet, theoretical considerations show that this kind of point defect hardly affects the electronic properties of a thin-film cell at all.” Thanks to neutron scattering, this finding, which is certainly reassuring for the manufacturers, has at last been experimentally confirmed. The results of Prof. Marcus Bär make it very clear that there is still a long way to go towards understanding the

materials used in photovoltaics, and that this is a major challenge. When studying a compound of copper, zinc and sulphur ( $\text{Cu}_2\text{ZnSnS}_4$ ), the head of the HZB junior research group “Boundary Design” discovered discrepancies between experiment and theory. “Thin-film solar cells based on this material system, which consists of especially abundant elements and thus promises another drop in costs, have undergone rapid development in recent years and already reach efficiencies above ten percent these days,” Bär says. After a great deal of effort, the field of solar energy research has managed to produce unusually phase-pure samples out of this material.

To analyse its electronic properties, the junior researchers scrutinised it with X-rays. From the absorption curve of relatively low-energy, “soft” X-rays and the resulting fluorescence emission, they could trace the so-called band structure – the energy distribution of electrons in the semiconductor material. “We found agreements with calculated diagrams of the band structure,” Marcus Bär reports. “Yet, some of our measurement results did not match the model calculations.” This surprising result whacks the ball back into the theoreticians’ court. It is their turn now to adapt and refine their calculations based on the experimental results.

*rb*

Appl. Phys. Lett. 98, 091906 (2011); (DOI: 10.1063/1.3559621): Comprehensive insights into point defect and defect cluster formation in  $\text{CuInSe}_2$ , C. Stephan, S. Schorr, M. Tovar & H.-W. Schock

Journal of Materials Research (2012), (DOI: 10.1557/jmr.2012.59):  $\text{Cu}_2\text{ZnSnS}_4$  thin-film solar cell absorbers illuminated by soft X-rays, M. Bär, B.-A. Schubert, B. Marsen, R.G. Wilks, M. Blum, S. Krause, S. Pookpanratana, Y. Zhang, T. Unold, W. Yang, L. Weinhardt, C. Heske & H.-W. Schock

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## SHAPE DETERMINES FUNCTION

Three-dimensionally imaged **catalyst particles** can optimise fuel cells.

The image Roman Grothausmann assembled using electron tomography and a processing algorithm looks kind of like a poodle that has rolled about in coloured candy. It shows a carbon surface bespeckled with nanoparticles. The image delivers unique insights into the three-dimensional structure of these particles, rendered in colour by a computer program. To generate the image, Grothausmann and colleagues from the Bundesanstalt für Materialforschung und -prüfung (BAM) made their own improvements on the method of electron tomography.

The ruthenium particles the physicist from the Institute for Applied Materials Research examined have long been used in solar fuel research at the institute. As catalysts they play an essential role in many chemical processes for industrial production, low-emission cars and eco-friendly energy conversion in fuel cells. To increase the efficiency of these processes, the particles need to be optimised: "It is especially important to understand the surface of the particles, since that is where the crucial chemical reactions take place," explains Grothausmann. As part of his doctoral thesis, he studied together with his colleagues ruthenium particles in particular, which will replace the expensive platinum in future fuel cells and could give new impetus to the technology. The particles are applied onto various carbon-based substrates as a ruthenium-chloride solution. The experiments are done in an electron microscope equipped with a sample holder that can tilt the sample by 140 degrees. Pictures from various angles are calculated into an overall three-dimensional image using a reconstructive algorithm developed by researchers working with Axel Lange at BAM.

### Insights from the particles' shape

Roman Grothausmann and his colleagues researching into solar fuels are interested in the shape and texture of the particles as well as how they rest on the substrate. All of these factors influence their efficiency as catalysts. To analyse them, the particles are divided into four different shape classes and coloured accordingly: blue for spheres, green for smarties, red for cigars and yellow for everything



Catalyst nanoparticles (coloured) adhere to a substrate (grey), captured by electron tomography. Novel processing algorithms were used for this three-dimensional image.

else. The particles' exposed surface is the only part that contributes to the desired reactions, so it is measured separately. Yet, for a fuel cell to deliver electrical energy with highest efficiency, the particles need more than just a large reactive surface; they also need a large enough contact area with the substrate. Only then can the generated charge carriers be easily conducted away as useful electricity. To find out whether an optimum size of contact surface exists in the competition between these two needs, Grothausmann is also analysing how the particles rest on the substrate.

The researchers use the three-dimensional imaging to make images of many particles at once, thereby delivering statistically significant results – on the size distribution of the particles and their preferred shapes, for example. The latter also influences the catalytic activity of the particles because the decisive atomic structure of the surface changes with its shape. Although the method does not resolve the particle surfaces to atomic precision, their shape alone provides plenty of information for drawing meaningful conclusions.

*ud*

J. Am. Chem. Soc., 2011, 133 (45), pp 18161–18171 (DOI: 10.1021/ja2032508): Quantitative Structural Assessment of Heterogeneous Catalysts by Electron Tomography, R. Grothausmann, G. Zehl, I. Manke, S. Fiechter, P. Bogdanoff, I. Dorbandt, A. Kupsch, A. Lange, M. Hentschel, G. Schumacher, J. Banhart

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## HARVESTING LIGHT UNDER BONNETS

Novel solar cells featuring **three-dimensional nanostructures** unify the advantages of different cell types. Researchers at HZB have created the basis for producing them.

Solar cells made from crystalline silicon dominate the photovoltaic market. Their appeal lies in the relatively high yield of electricity which they generate from sunlight – industrially produced cells achieve around 16 percent efficiency. Yet, they are relatively thick, they require a lot of materials to produce, and it is difficult to connect individual cells into large modules. By contrast, thin-film solar cells, which feature a thin film of semiconducting material applied only a few hundred nanometres thick over a large area of a glass plate, for example, are cheaper to produce and easier to combine into modules. Their efficiency, however, is lower.

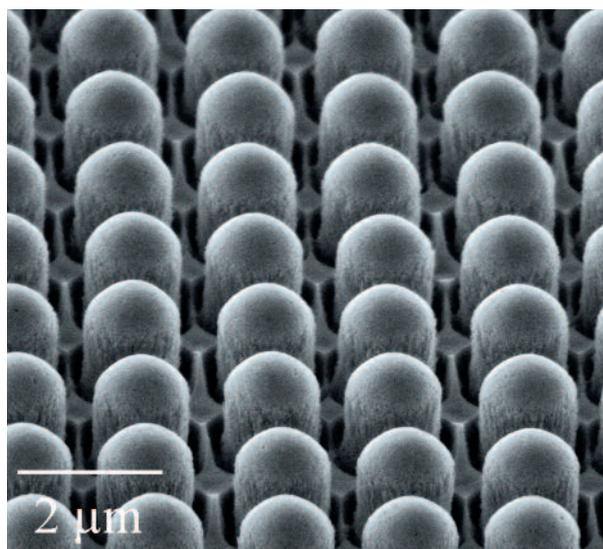
Dr. Tobias Sontheimer, physicist at the Institute for Silicon Photovoltaics of HZB, has now succeeded in bridging the gap between the two technologies, unifying the advantages of crystalline and thin-film solar cells: He has developed a method for quickly and easily applying tiny, regularly arranged silicon crystals as a thin layer onto a substrate. “So far, the typical method is to deposit thin layers of silicon using chemical gas phase deposition,” says Sont-

heimer. This complex process takes several hours. The Berlin researchers instead used an electron beam to vaporise the semiconducting material from a molten mass and apply it onto a glass plate. “It took less than a minute to produce a thin silicon layer this way,” he says. Sontheimer and research colleagues at HZB then tweaked the method for producing solar cells. The crystalline layer produced by this method is equal in quality to that of a silicon layer produced by conventional means – the prerequisite for efficient energy conversion within a solar cell.

### Periodically organised silicon crystals

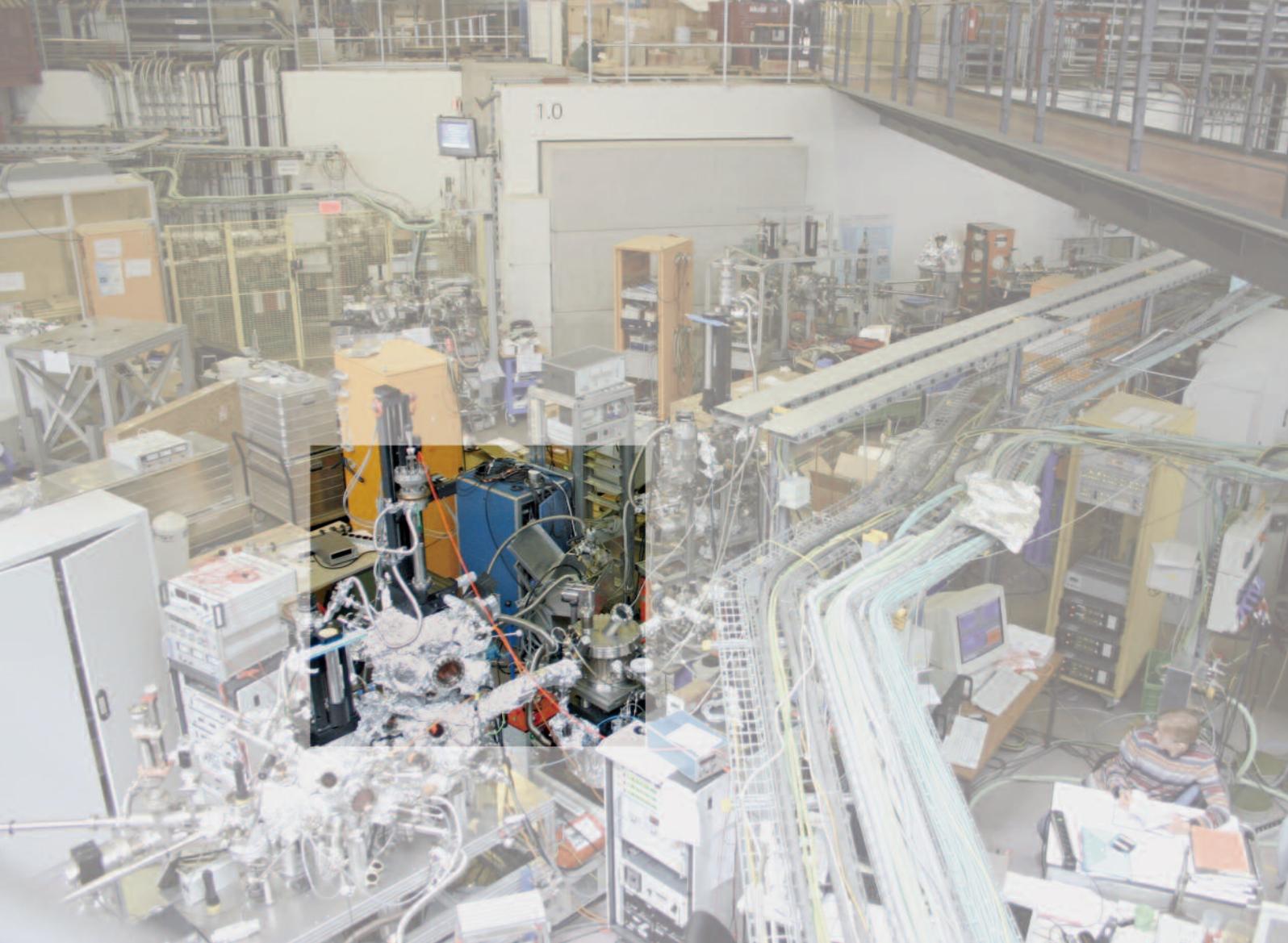
“To achieve high efficiency, you also need a lot of light to be absorbed into the cell,” explains Tobias Sontheimer. This can be ensured by creating a texture on the silicon. Up to now, this could only be done by roughening the surface, which is an imprecise and difficult method to control. Working with Schott AG in Mainz, Sontheimer, on the other hand, managed to produce a periodic arrangement of identically shaped silicon crystals. They did so by transferring a 3D texture onto the surface of a glass substrate using a stamp fashioned with a nanometre-fine structure. “The next step was to coat with silicon,” says Sontheimer. Depending on how this landed on the substrate, it produced ordered (crystalline) or unordered (amorphous) areas, where the amorphous areas were subsequently etched off. This left behind a “forest” of filigree bonnets of crystalline silicon: “light catchers” in which sunlight becomes well and truly trapped.

The method allows for controlled variation in the size and spacing of the silicon bonnets – over a wide range from nanometres to microns – so that they can be adapted to absorb specific wavelengths of light. The HZB researcher has thus laid the foundation for designing novel, high-efficiency, three-dimensionally structured thin-film solar cells. Tobias Sontheimer intends to produce the first cells of this kind very soon. *rb*



Microscopic view of silicon bonnets on a thin-film solar cell. The spacing of the bonnets is just under two thousandths of a millimetre.

Energy Procedia 10 (2011) 61 – 65 (doi:10.1016/j.egypro.2011.10.153): Polycrystalline silicon thin films by high-rate electron-beam evaporation for photovoltaic applications, C. Becker et.al.



## JOINT VENTURES

**H**elmholtz-Zentrum Berlin cooperates closely with numerous German and international universities, research institutes and companies.

These cooperatives are formed in the interest of pure research, knowledge exchange and the joint use of facilities and methods. Knowledge exchange is the primary purpose, for example, of the transatlantic solar research cooperative HZB has entered into with two other German research establishments and the U.S. National Renewable Energy Laboratory (NREL). Their aim is to expand the scientific basis behind renewable energy supplies.

HZB scientists working with colleagues from the Swedish University of Lund have taken an important step towards a

stable free-electron laser (FEL). The synchrotron light now produced at unmatched quality opens up new possibilities in structural and materials research.

The properties of complex materials, which can be used in a targeted manner in all kinds of applications, are governed by mutually influencing factors such as charge, spatial arrangement and spin. Further research into the principles of action of such materials is the goal of the new virtual institute “Dynamic Pathways in Multidimensional Landscapes” at HZB, involving colleagues from Helmholtz-Zentrum DESY and other partners.

Read more about HZB’s cooperatives over the following pages.

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## FASTER MARKET MATURITY FOR INNOVATIONS

HZB and two other research centres of the Helmholtz Association and NREL of the U.S. are now collaborating on **solar energy research**.

**H**elmholtz-Zentrum Berlin, the German Aerospace Center (Deutsches Zentrum für Luft- und Raumfahrt, DLR) and Forschungszentrum Jülich (FZJ) are the three leading centres for solar energy research within the Helmholtz Association. HZB and FZJ are primarily studying new thin-film materials for solar cells. DLR is one of the world's leading institutes for the development of solar thermal power plants. DLR researchers are also working on novel storage media for solar thermal power plants, to store daytime heat into the night-time hours so they can generate electricity continuously around the clock. In June 2011, the three institutes signed a Memo of Understanding (MOU) with the National Renewable Energy Laboratory (NREL) in the field of solar energy research. NREL is the largest American research institute for renewable energies and works on behalf of the U.S. Department of Energy. The institute has been developing solar thermal power plants for many years.

### Exploiting the full potential of solar energy

With the cooperative signed at the American embassy in Berlin, the partners involved intend to close the gaps in research and accelerate the development of technologies. "This agreement unites two powerful organisations that can achieve even faster progress in the fields of photovoltaics and solar heating through joint research projects," Professor Jürgen Mlynek, president of the Helmholtz Association, emphasises. "The agreement promises to advance the state of knowledge and the development of new materials and technologies that will form the basis of next-generation solar cells and solar fuels," says NREL director Dr. Dan Arvizu.

Studies demonstrate the enormous potentials of solar energy. The deserts of the earth, for example, pick up more solar energy in six hours than all of humanity consumes in



Transatlantic cooperative (left to right): Prof. Dr. Bernd Rech, Prof. Dr. Anke Kaysser-Pyzalla, Dr. Ulrich Breuer (all HZB), Dr. Dan Arvizu (NREL), Prof. Dr. Robert Pitz-Paal (DLR), Thomas Rachel, Prof. Dr. Harald Bolt (FZJ), Prof. Dr. Jürgen Mlynek, Greg Delawie and Prof. Dr. Uwe Rau after they have signed the treaty.

a year. But, if solar energy is to be converted cost-effectively into electricity, solar cells and solar thermal power systems will have to operate more efficiently and be cheaper to build.

### Converting sunlight into energy carriers

Scientists at the research centres involved are improving the technologies we already use today. At the same time, they are pursuing new avenues. For instance, they are developing solar direct steam generation technology (DSG) to market maturity for solar thermal power plants. In this type of solar power plant, concentrated sun rays directly generate water steam, which ultimately drives an electric generator. In photovoltaics, HZB researchers are working on approaches using nanostructured thin-film materials and novel module architectures. "This cooperative will make a significant contribution to overcoming the challenges in developing new, even higher performance solar cells," stresses scientific director of HZB, Professor Dr. Anke Kaysser-Pyzalla. "We will be concentrating on researching into new materials for photovoltaics and developing concepts for directly converting sunlight into chemical energy carriers."

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## JOINT RESEARCH IS PROLONGED

The **Russian-German Laboratory** at the electron storage ring BESSY II celebrated its tenth anniversary in 2011.

Scientific cooperatives between Germany and Russia have been funded at the highest political level for many years. The basis is the 'Joint Declaration on a Strategic Partnership in Education, Research and Innovation', the aim of which is to expand cooperation over a broad spectrum of research areas. The latest example of this cooperation was the 'German-Russian Year of Education, Science and Innovation 2011/2012', for which many events were held in both countries between July 2011 and June 2012. Russia is also an important partner for the Helmholtz Association in helping to establish the international accelerator centre FAIR at GSI Helmholtzzentrum für Schwerionenforschung (heavy ion research) in Darmstadt. Another good example of successful cooperation is the Russian-German Laboratory at the electron storage ring BESSY II of Helmholtz-Zentrum Berlin, which celebrated a decade of existence in June 2011 with a workshop in Berlin. In the scope of this unique cooperative between German and Russian scientists, HZB, Freie Universität Berlin, Technische Universität Dresden, St. Petersburg State University, the Ioffe Institute in St. Petersburg and the Kurchatov Institute and Shubnikov Institute of Crystallography in Moscow are working together to develop instruments and methods at the synchrotron radiation source.

### Further expansion planned

In the centre of the Russian-German Lab is a beamline for soft X-rays for researching the atomic structure of matter. "Experiments with synchrotron radiation play an important role in pure research," says Eckart Rühl, professor of physical chemistry at Freie Universität Berlin and chairman of the steering committee of the Russian-German Laboratory. Because the demand from researchers who want to use the beamline to study material samples exceeds the free measurement time, the research capacities shall be expanded in future. The German Federal Ministry of Education and Research (BMBF) provides funding for stocking up on experimental resources. In the planning is a so-called undulator beamline with which the Russian-German Laboratory shall be expanded into a worldwide leading measuring station for angle and spin resolved photoelectron spectroscopy over the next two years. The aim is to study magnetic materials of nanoscale dimensions.

### Numerous successes

The Russian-German Laboratory can look back on ten years of diverse scientific successes: Research at the laboratory has issued more than 250 publications in prestigious journals. 48 diploma theses, 14 doctoral theses and



Left to right: Prof. Dr. Andrei G. Zabrodskii (Ioffe Institute), Prof. Dr. Clemens Laubschat (Technische Universität Dresden), Prof. Dr. Sergey Tunik (University St. Petersburg), Prof. Dr. Anke Kaysser-Pyzalla (HZB) und Prof. Dr. Peter-André Alt (Freie Universität Berlin) signed the contract extension for the Russian-German Laboratory as representatives of their institutes.

two habitations are largely based on results obtained here. Research at the Russian-German Laboratory is supported by the International Centre of Excellence for Natural Sciences established by the St. Petersburg State University and Freie Universität Berlin in 2010. This German-Russian Interdisciplinary Science Centre (G-RISC) is based in St. Petersburg and is financed by the German Academic Exchange Service (DAAD) from funds of the Federal Foreign Office. G-RISC provides a scientist who, in the employ of FU Berlin, supervises the users of the German-Russian Laboratory at BESSY II on their experiments.

#### New agreement is being prepared

So that joint research can continue, the partners involved in the German-Russian Laboratory used the workshop as an occasion to prolong the agreement to the end of 2013. Once the new beamline at Undulator U125/2 funded by the BMBF goes into operation at BESSY II, which is planned for mid 2013, a new agreement will have to be finalised. This agreement is already being prepared at Helmholtz-Zentrum Berlin and shall be negotiated with the partners involved in 2012.

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## ON THE PATH TO A STABLE FREE ELECTRON LASER

Scientists from HZB and the synchrotron radiation source MAX-lab are cooperating to produce **synchrotron light** at unparalleled quality.

Researchers such as Dr. Johannes Bahrtdt of HZB are striving to improve synchrotron light in order to push the door of science wider open for ever more detailed insights into matter. The development of stabilised free electron lasers (seeded FEL) is considered an important milestone along this road, since these lasers can combine high intensities of short wavelength light with the spectral purity and temporal coherence of light packets. So far, this combination has only been possible at the longer wavelengths from infrared to UV. In cooperation with colleagues from the Swedish University of Lund and together with other scientists from HZB, Bahrtdt performed a so-far unique experiment at the synchrotron radiation source MAX-lab in Lund. The researchers succeeded in generating coherent light pulses in the extreme vacuum ultraviolet spectral region of 66 nanometres that are only 200 femtoseconds long and exhibit variable polarisation. Thus, they reproduced the stability and evenness of a commercial laser of longer wavelength in a light pulse within a scientifically significant spectral region in which no light sources of this quality have existed before. "We thus took an important step towards a stable free electron laser," says Dr. Johannes Bahrtdt, head of the Undulators Department at HZB. For the experiment set-up in the scope of the EuroFEL Design Project, HZB's Undulators Department developed the entire undulator system for the experiment – that is the



Dr. Johannes Bahrtdt heads the Undulators Department at HZB.

linear arrangement of dipole magnets used to produce the synchrotron radiation – which they then transported to and installed in Sweden. They also provided the fibreglass systems for electron beam diagnostics. To maximise the coherent radiation, the electron bunches must be compressed in the direction of flight. The HZB institute Methods and Instrumentation for Synchrotron Radiation Research contributed a terahertz detection system for optimising the bunch compression in the injection linac. The colleagues from Sweden set up the pre-accelerator as well as two new laser systems at MAX-lab with which the electrons were produced and modulated. Furthermore, they set up the necessary photon diagnostics to demonstrate the transverse coherence and reduced energy bandwidth of the coherent radiation.

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## EXPLORING THE GOVERNING PRINCIPLES OF FUNCTIONAL MATERIALS

The Helmholtz Association is funding the new **virtual institute** “Dynamic Pathways in Multidimensional Landscapes” at HZB. In turn, HZB is involved in another virtual institute of its own.

**V**irtual institutes are an instrument the Helmholtz Association uses to initiate and consolidate cooperatives between universities and Helmholtz centres. They are financially supported by the Helmholtz Association and partners involved for a period of up to five years before a decision is made about continuing the cooperation. The aim is to reinforce university research by establishing visible competence centres and networking with centres of the Helmholtz Association, the largest scientific

man and international partners, the Helmholtz centres HZB and DESY are studying the overarching principles of complex materials.

The properties of complex materials are governed by coupled internal degrees of freedom, i.e. orbital, spin, lattice and charge, which interact with the environment. These interactions give rise to functionalities that can be utilised and even tailored, say, to obtain energy, to store data or to communicate. The scientific program is therefore oriented on

the four dimensions of energy, time, space and momentum and intentionally crosses the conventional disciplinary boundaries between solids, liquids and molecular systems. The virtual institute’s instrumentation is based on the accelerator-based X-ray sources of HZB and DESY, the Linac Coherent Light Source in Stanford and the laser-based instruments at the universities involved.

### Multifunctional biomaterials for medicine

The institute “Multifunctional Biomaterials for Medicine” is another Helmholtz virtual institute that opened at the end of 2011, where HZB is one of the core partners alongside the leading Helmholtz-Zentrum

Geesthacht in Teltow (HZG) and FU Berlin. Its mission is to study the interactions between proteins and polymeric biomaterials, which have never been fully understood or controllable. Modern concepts for medical therapies, however, frequently rely on such multifunctional biomaterials. These could be implant materials used inside the body, perhaps, or active agent carriers. Trouble arises when interactions between the body’s proteins and these biomaterials fundamentally modify the properties and behaviour of the materials. With this virtual institute, FU Berlin and the two Helmholtz partners intend to reinforce their networking and work on this research topic over the coming years, together with further partners from Germany and abroad. *cn*



HZB provides research facilities for the new virtual institute at its electron storage ring BESSY II.

organisation in Germany. Virtual institutes shall serve as the hub for future, larger strategic research projects, allowing strong international networking while promoting junior scientists.

### New virtual institute at HZB

At the end of June 2011, the Helmholtz Association gave HZB the go-ahead for the virtual institute “Dynamic Pathways in Multidimensional Landscapes”, one of twelve virtual Helmholtz institutes in total that were newly established in 2011. The Helmholtz Association is funding the institute with a total of 2.29 million euros. At the new virtual institute, together with FU Berlin, TU Berlin and other Ger-

## PVCOMB IS A PREFERRED PARTNER FOR INDUSTRY

PVcomB, an initiative of HZB and TU Berlin, was officially inaugurated in 2011 and has already entered into several **cooperatives** with industry.



The Competence Centre Thin-Film- and Nanotechnology for Photovoltaics Berlin (PVcomB) has dedicated itself to the transfer of knowledge from the laboratory to applied industrial processes regarding all aspects of thin-film photovoltaic technology and product manufacture. The full subsidiary of HZB was officially inaugurated on 30 March 2011 in the presence of BMBF state secretary Dr. Georg Schütte and president of the Helmholtz Association Prof. Dr. Jürgen Mlynek. “The rapid expansion of renewable energies will require investments if we are to improve the technologies even further and make them economically competitive. Pho-



Dr. Rutger Schlatmann (left), Director of PVcomB, welcomes the guests to the official inauguration of the establishment.

tovoltaics has a great potential here, especially if we succeed in reproducing the progress achieved in the laboratory as marketable products. PVcomB has taken a step in this direction,” says Prof. Dr. Jürgen Mlynek.

At the inauguration, Wista Management GmbH, operator of the technology park Berlin Adlershof, held the ground-breaking ceremony to mark the start of construction of the new Centre for Photovoltaics, ZPV. Starting in 2013 as a direct neighbour to PVcomB, a strong contributor to the Solar Cluster Adlershof, ZPV will provide a total of 8,000 square metres of production, laboratory and office space for small and medium-sized companies of the cleantech sector. More than 1000 jobs in photovoltaics have been created in Adlershof over the last four years.



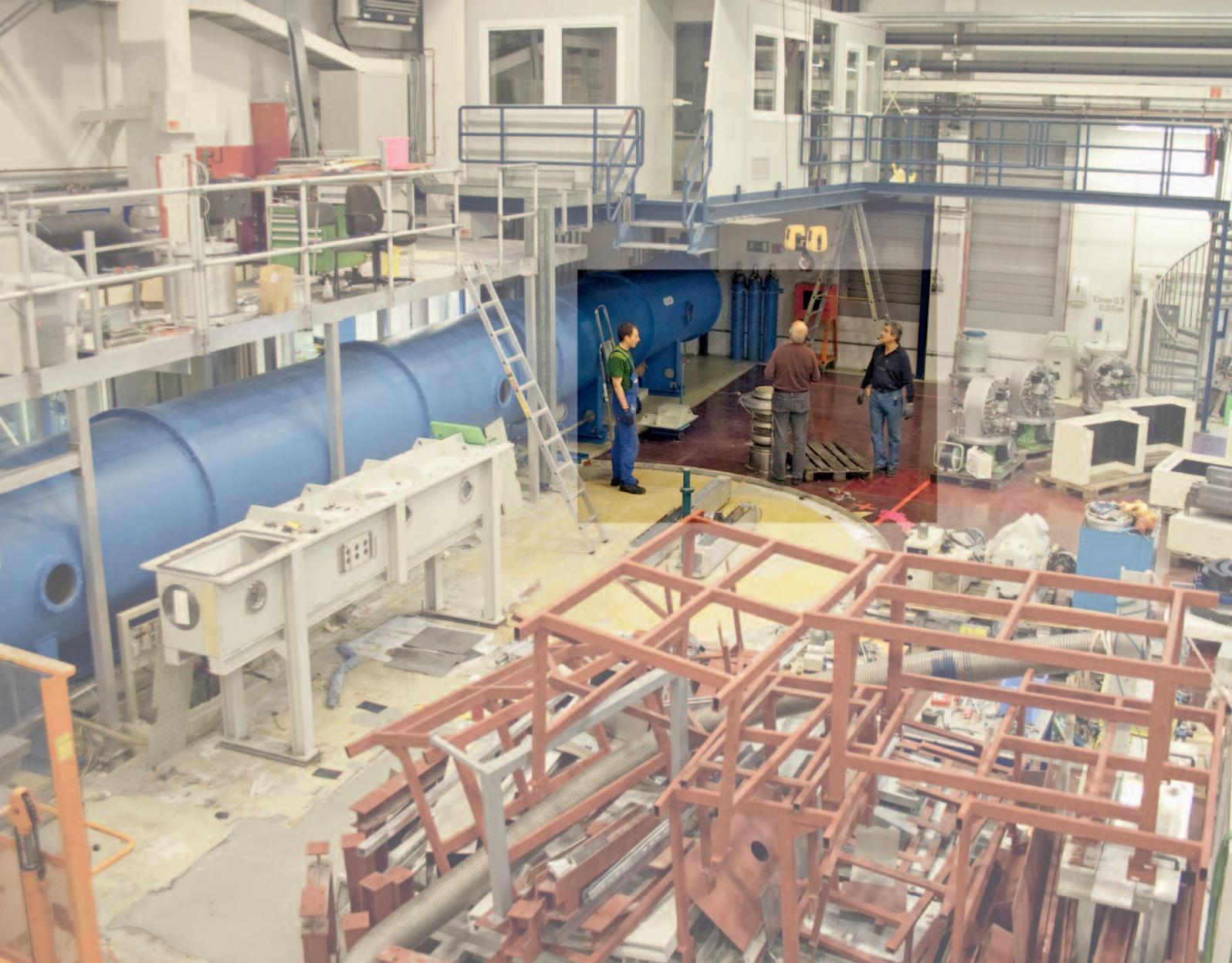
Modern production line for solar cell modules at PVcomB.

PVcomB, established in 2007 by HZB and TU Berlin together, already began working before its inauguration and produced its first photovoltaic modules by the end of 2010. To improve the technology transfer along the value-added chain of the photovoltaic industry, PVcomB has been cooperating with several industrial partners since 2011 in the scope of the Innovation Alliance Photovoltaics initiated by the BMBF and the BMU. With the associated partners Inventux, NEXT ENERGY and Hüttinger Elektronik, PVcomB is starting a research project to increase the efficiency in micromorphous technology to above twelve percent while also doubling the deposition rate. Another project shall be implemented at PVcomB together with Leybold Optics.

### Cooperation agreement with centrotherm photovoltaics

In the thin-film module segment, PVcomB signed a cooperation agreement in mid April 2011 with centrotherm photovoltaics AG, a world leading technology and equipment supplier for the photovoltaics sector. Together with PVcomB, the company intends to advance the process technology for manufacturing thin-film modules. Prof. Dr. Hans Werner Schock, one of the initiators of PVcomB and director of the Institute of Technology at HZB, sees major opportunities in the cooperative: “Firstly, the facilities of centrotherm photovoltaics AG give us an established platform for working on questions from industry relating to research and development. Secondly, on this basis, we will be able to scale up new developments from pure research to an industry-scale line as quickly as possible.” The cooperative offers the added advantage of improving the education of engineers and scientists at these high-tech facilities.

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## FUTURE PROJECTS

With the neutron source BER II and electron storage ring BESSY II, Helmholtz-Zentrum Berlin operates two large-scale facilities that scientists from around the world use for their experiments. The neutron source has undergone renovation and many improvements to continue offering attractive research stations in Berlin-Wannsee. Similarly, the electron storage ring is being updated in a series of important projects to reflect the latest advancements in science. In the scope of the *BERLinPro* project, for example, photoelectrons were produced and accelerated for the first time using a superconducting electron source

(SRF Gun). This was an important step towards developing an energy recovery linac that shall push the door open to new applications in science. Improving synchrotron radiation was also the aim of the Technology Centre for Ultra-precise Optical Gratings approved in 2011 by the Berlin Senate Administration for Education, Science and Research and funded by the EU. With the large-scale project *EMIL* (Energy Materials In-situ Laboratory Berlin), HZB is going to build a new X-ray beamline for analysing materials for renewable energy generation.

Read more about our future projects over the following pages.

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## MILESTONE FOR BERLINPRO



Scientists at HZB have produced and accelerated the first photoelectrons using their **superconducting electron source**, the SRF Gun.

**W**ith its feasibility study *BERLinPro*, HZB is testing whether a linear accelerator featuring energy recovery can be achieved in principle. Such a system could improve the capacity of synchrotron sources so greatly that, in addition to the excellent snapshots they can already take of their samples, researchers would also be able to follow the changes in the observed structures at millionths of a split second intervals.

In April 2011, in the scope of *BERLinPro*, the project group led by Dr. Thorsten Kamps of the Institute for Accelerator Physics in cooperation with the HZB Institute for SRF Sci-

ence and Technology succeeded for the first time in generating and accelerating an electron beam from a superconductive photocathode using a superconducting high-frequency photoinjector. This was an important step in the realisation of *BERLinPro*, since operating an energy recovery linac requires high-brilliance electron sources. Also involved in the planning, build-up and operation of the project were experts from the Jefferson Accelerator Laboratory (USA), the Polish computer centre IPJ Swierk, DESY and the Max Born Institute.

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## BETTER GRATINGS FOR BETTER LIGHT

Helmholtz-Zentrum Berlin is establishing a technology centre for **ultraprecise optical gratings**. It will improve the efficiency of synchrotron radiation from BESSY II.

**I**rradiating samples with the short-wave X-ray light from BESSY II is an excellent way to gain unique insights into the structure of matter. If scientists are to make detailed analyses of their samples, then the light they shine onto them must be a known quantity. Accordingly, they start by fanning the X-ray light out into its individual wavelengths using diffraction gratings. The wavelengths needed for the respective studies are then filtered out and focused onto the samples. Helmholtz-Zentrum Berlin is establishing a technology centre for ultraprecise optical gratings specifically for producing and enhancing these diffraction gratings. It is being implemented by the new HZB Institute for Nanometre Optics and Technology and supervised by the workgroup of Dr. Bernd Loechel.

### Five million euros from ERDF

The Berlin Senate Administration for Education, Science and Research laid the foundation for this new project at the beginning of 2011 with a grant of five million euros from the European Regional Development Fund (ERDF).

“These means were used, among other things, to take over the existing precision grating production lines of Carl Zeiss AG and set them up in Berlin,” Loechel reports. At the new technology centre, the diffraction gratings shall be produced on these lines using improved equipment, and even enhanced to allow the highest light yield possible.

### First functional blazed gratings in 2012

To this end, the researchers are producing so-called blazed gratings, which feature an etched saw-tooth profile. Producing precise saw-tooth structures at a nanoscale, however, is a technological challenge. The first blazed gratings have already been experimentally produced using the Zeiss equipment installed at HZB. Since the environmental conditions have not yet been optimised, they have only produced gratings with 2000 lines per millimetre and 5 centimetres in length so far, but already at an equal quality to that of international suppliers. The technology centre expects to provide the first functional blazed gratings in the second half of 2012.

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## EMIL AND RESEARCH ON ENERGY MATERIALS

With the **large-scale project EMIL** (Energy Materials In-situ Laboratory Berlin), HZB shall create new opportunities for researching energy materials by the beginning of 2015



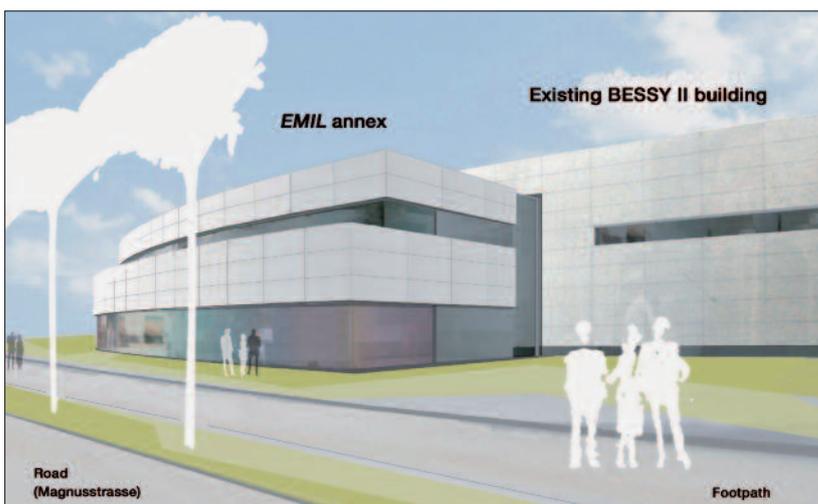
In 1929, children's book author Erich Kästner literarily associated the name Emil with Berlin for generations of readers to come. In 'Emil and the Detectives', the eponymous hero set out in the city on the Spree to track down and convict a thief with the help of his friends. With its large-scale project *EMIL*, HZB is on a search of its own, with the help of the Max Planck Society: The search for better materials for renewable energy generation. The project revolves around building a new X-ray beamline at the synchrotron source BESSY II for analysing such materials. Following the positive assessment of the project by an external committee of experts engaged by the scientific advisory board, the supervisory board of HZB gave the go-ahead for *EMIL* in October 2011. This worldwide unique laboratory, planned to commence operation at the beginning of 2015, is being built at BESSY II where materials for photovoltaics and photocatalytic processes can be studied by X-ray analysis. Several experimental stations will be established, giving researchers access to soft and hard X-rays for their research. "In the planned laboratory, we will combine material production with ultra-precise analysis of layer

properties better than anywhere else in the world without interrupting the vacuum needed for synthesis, allowing us to develop better thin-film solar cells and energy stores," says Dr. Klaus Lips, who is heading the project at HZB.

### Improving solar cells with Sissy

One of the three experimental stations to be established in the scope of *EMIL* is called Sissy (Solar Energy Materials In-Situ Spectroscopy at the Synchrotron). This ultra-modern analytical measuring station for solar energy research will help advance thin-film solar cells. To make solar electricity competitive, researchers are looking for new material combinations that will make solar modules cheaper and more efficient to produce and run. Solar cells consist of ultra-thin layers that convert incident solar energy into electric charge. The boundaries between these structures are only a few atomic layers thick, but they determine the chemical-electronic properties of the solar cell. The solar cells are studied using the spectroscopic method of X-ray analysis. However, the existing measuring stations for this method are often booked out months in advance.

The Solar Cell In-Situ Laboratory at the Synchrotron (Sissy) will be connected directly to the electron storage ring BESSY II and will give researchers daily exclusive access to X-ray analytics. At Sissy, researchers will no longer need to transport silicon layers of solar cells to the measuring station, rather they will be able to prepare them directly in situ in the so-called cluster tool. Researchers will thus be able to study their samples in situ at the Sissy lab, i.e. while they are growing, or to transfer them directly from the preparation chamber into the analysis chamber. This will maintain the vacuum crucial for such analyses, since the outer and boundary surfaces change under the influence of air. Another special feature of Sissy is that it will provide 'hard' and 'soft' X-rays for the first time in a single laboratory. A refined system will bring the separately generated



The laboratory *EMIL* is being built onto the electron storage ring BESSY II for 2015. It will give solar energy researchers additional measuring stations for their samples.

beams together at SISSY. 'Soft' X-rays are for precise analysis of surfaces. Energy-richer 'hard' X-rays give additional information about the deeper layers. Using both, researchers can therefore make a comprehensive study of boundary surfaces. SISSY will also be connected to an X-ray photoemission electron microscope (XPEEM), which will allow imaging of nanoscale characteristics of the boundary surfaces.

### Measuring station for catalysts

Research into catalysts will take place at another measuring station, CAT@EMIL. This is being funded and built by the Max Planck Society. Both measuring stations are primarily intended for in-house research, while one third of the measurement time will be made available for external users from universities and industry. The third measuring station

planned in the *EMIL* project (60to6), which has received no funding as yet, would be primarily dedicated for external users, at about 80 percent of the measuring time.

Building *EMIL*, with its analytical tools SISSY and CAT, is costing around 18 million euros. HZB will invest 6 million euros in *EMIL* and the Max Planck Society will participate with a further 6.7 million euros. The German Federal Ministry for Education and Research (BMBF) is funding construction of SISSY with 5.7 million euros from the 'Innovation Alliance Photovoltaics'. "Realising *EMIL* together with the Max Planck Society, and creating the best analytical conditions for researchers worldwide, would have been inconceivable without the fusion of the Hahn-Meitner Institute and BESSY in 2009. The new *EMIL* project makes the benefits of the merger especially clear," says Prof. Dr. Anke Kaysser-Pyzalla, scientific director of HZB. cn

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## RESEARCHING UNDER EXTREME CONDITIONS

For 2013, HZB is building a **high-field magnet** with an initial field strength of 25 Tesla. The neutron instrument "extreme environment diffractometer" (EXED) is especially designed to work with it.



Completion of the world's strongest high-field magnet (HFM) for neutron scattering experiments will create unique experimental conditions. The HFM is being structurally adapted to give the neutrons undisturbed access to the samples placed inside the magnet for study. Lying in the centre of the HFM is a double-funnel. The neutron beam enters on one side and strikes the sample at the narrowest point of the funnel. The neutrons scattered by the sample escape out the other side of the funnel, where they are measured by detectors. There is one slight disadvantage to this arrangement, however: "Without high magnetic fields, the scattered neutrons can be observed in all directions, but the double-funnel only allows this at an angle of 30 degrees," technical project manager Dr. Peter Smeibidl explains the problem.

With the EXED, one of about 15 instruments at the neutron source BER II, the scientists compensate for this problem. With its multispectral neutron guides, EXED allows experimenters to use an unusually large bandwidth of different neutron wavelengths. Accordingly, instead of using only a single wavelength, the sample can be bombarded with neutrons of varying wavelengths from 0.5 to 15 Ångström and their scattering recorded. This data gives the scientists meaningful insights into the constitution of the sample.



At the end of the neutron guide (right) are two detectors, one for forward and one for backward scattering. In the middle of the instrument (centre) is an instrument table for test measurements. This is where the substantially larger magnet system will later be. Also installed in the course of the neutron guide are various chopper systems (not pictured) as well as computer systems for controlling the chopper systems and analysing the detector data.

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## CANDLELIGHT TURNS TO FLOODLIGHT

The **European Spallation Source (ESS)**, to be built on Swedish soil starting in 2013, will provide unique insights into matter. HZB has many scientists involved in the planning.

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**N**eutrons offer researchers a unique view into the inner workings of matter and are now an indispensable tool of pure research. They render visible structures and motion processes at atomic scales. The more intense the neutron beams, the more details measurements can reveal about the make-up of matter. The scientists at HZB have recently upgraded the neutron source BER II and its instru-

mentation to be able to fire even more neutrons at samples in future.

Looking further into the future, neutron sources will almost exclusively generate neutrons by spallation, that is by fragmentation of nuclei. To do so, scientists first accelerate protons, the positive particles of atomic nuclei, to near light speed. These protons are then redirected onto a heavy metal



The European Spallation Source (ESS) is still only a computer simulation, yet the scientists at HZB are already involved in planning the design and instrumentation of the facility to be built in Lund, Sweden.

block of tungsten. The high-energy protons collide with the atomic nuclei in the metal block, exciting them to such a high energy state that a large number of neutrons “steam off”. This effect produces the most scientifically useful neutrons per unit energy of waste heat. Because neutrons freshly produced by this method are too fast and energy-rich, they have to be braked before they can be used for research. This is done using moderators containing methane or hydrogen arranged closely around the metal block.

### Europe’s largest neutron source

With the construction of the European Spallation Source (ESS) in Lund, a small university city near the port city of Malmö in the south-west of Sweden, this method shall be applied on a large scale. 17 European countries are involved in this project, which is still in the design-update phase for the instrumentation and components. Construction of ESS is planned to commence in 2013 and the first neutrons shall fly in 2019. From 2025, the facility shall be fully operational and available to users from around the world. The total costs for planning, construction and operation of ESS are estimated at 1.5 billion euros.

ESS will deliver pulsed neutron packets for researchers to perform their experiments. “While the neutron flow obtained by nuclear fission is continuous, the ESS will deliver neutrons as bundled packets that will strike the sample at short intervals,” explains Dr. Klaus Habicht, head of the department Research with Spallation Neutrons at HZB and project manager of HZB’s work in the design-update phase of ESS. In each packet – scientists speak of neutron pulses – neutrons exist at various speeds. They are redirected through beam tubes to the various experimental stations with their specific instruments. Time-of-flight experiments that use neutron pulses by design benefit especially from pulsed sources, since the neutrons are already portioned into packets at the very source.

### HZB’s involvement in construction

ESS will deliver especially long neutron pulses, with which the neutron intensities can be further boosted to the benefit of the experiments. Metaphorically speaking, if researchers have been examining materials under candlelight so far, then the neutrons at ESS will provide the brilliance of a floodlight. They will allow researchers to pursue complex questions in any field, including physics, biology, chemistry and medicine, working with the tiniest quantities of material. New methods that have been largely developed at HZB will allow optimal use of the long pulses. For the experimental verification of new concepts and prototypes of critical instrument components, HZB has built a dedicated testing beamline that simulates the temporal structure and repeat frequency of the pulses at ESS. HZB makes these facilities available to scientists of the joint project and of ESS.

### Developing instruments for ESS

Until ESS fires up in 2019, the researchers at HZB still have much to do. From two decades of in-house research and supervision of external users as instrument operators at the neutron source BER II, they know exactly what researchers need and they are contributing this expertise to the project. How a neutron instrument is built depends namely on what questions the scientists intend to study. Under the direction of Dr. Klaus Habicht, the HZB colleagues are developing instrument concepts mainly for studying thin-film systems (reflectometry), for imaging large objects (tomography), for motion processes of atoms and molecules (time-of-flight spectroscopy) and for studying matter under extreme conditions, such as extremely high magnetic fields. A group formed specifically in the Research with Spallation Neutrons Department is helping optimise the instrument concepts by running simulations in the software VITESS developed at HZB specifically for this purpose. cn

## HIGH-TECH DETECTORS FOR ESS

One of the key technologies that will allow optimal use of the high neutron flow at the new spallation source is efficient detector systems that can be run without helium-3. This is precisely what the colleagues at the detector lab are working on in the “Detectors” work package. One instrument class (reflectometry and imaging) requires detectors with a spatial sensitivity of around 100 to 500 µm, since the precisely measured spatial distribution of the neutrons harbours information of great interest to science. At the same time, the detectors must be able to capture at count rates in excess of 10<sup>7</sup> neutrons per second, and save these onto computers via readout electronics. For this, the workgroup led by Dr. Thomas Wilpert is develop-

ing so-called MSGC (multi-strip gas chamber) detectors. These use thin layers of gadolinium-157, one of the rare earth elements, to capture the neutrons with particularly high efficiency. The secondary electrons produced are amplified at the MSGC electrodes and read out using the latest chip technology. Two-dimensional spatial information is finally calculated in a so-called FPGA (field programmable gate array), a component that allows greatest flexibility and which no modern data acquisition set-up should be without. The results regarding detection efficiency and spatial and temporal resolution will help decide what components shall ultimately come into use at ESS.

## HONORARY CHAIRMAN OF TOYOTA VISITS HZB

**DR. SHOICHIRO TOYODA** GATHERS INFORMATION AT BESSY II ABOUT A JOINT GERMAN-JAPANESE RESEARCH PROJECT.

HZB and the Cluster Research Laboratory of the Toyota Technology Institute (TTI), a technical university located in Nagoya east of Tokyo and founded in 1981 by Toyota, have been working together for the last two years on joint research topics. Besides HZB and TTI, also part of the team are the Technische Universität (TU) Berlin and the



Dr. Shoichiro Toyoda (left) on his visit to HZB.

University of Freiburg. The main topic of the researchers is how can exotic magnetic phases be stabilised on an atomic scale by controlling their size and composition? The results could lead to a better understanding of the fundamental magnetic properties of materials that are of paramount importance to industry. To this end, the scientists set up an experiment at BESSY II where a superconductive magnet in the Japanese TTI laboratory and a cryogenic ion trap developed at HZB, the TU Berlin and the University of Freiburg, are used. This ion trap makes it possible to investigate tiniest particles of only a few atoms in size with an X-ray beam from BESSY II and moreover allows a detailed insight into the magnetic structure of such particles. In December 2010, the ion trap was used for the first time to investi-

gate the components involved in the magnetisation of ultra-small particles. Against this background, Dr Shoichiro Toyoda – a son of the founder of the Toyota company, who himself was president of the Japanese automotive multi from 1992 to 1999 – visited the electron storage ring BESSY II in October 2011 together with a delegation of experts. Dr Tobias Lau from the Institute for Methods and Instrumentation of Synchrotron Beam Research initiated and prepared the visit and, together with his Japanese cooperation partner – Professor Akira Terasaki from TTI – informed the guests about the current status of the research project. Dr Toyoda's visit to HZB endorsed the excellent reputation that the research projects at HZB as part of the cutting-edge research in Germany enjoy around the world.

## GERMAN CHANCELLOR HONOURS HZB TRAINEES

KATRIN KERRMANN, WHO TRAINED AT HZB TO BE A **MECHATRONICS ENGINEER**, WAS HANDED HER DIPLOMA BY NONE OTHER THAN ANGELA MERKEL.

The sandwich course system with its intermeshing of practical training modules in companies and theoretical training modules in vocational colleges is still held in high esteem. And to underline this, the German chancellor Angela Merkel came in September 2011 to inaugurate the new training year in the ABB training centre in Pankow, Berlin. Within the framework of the ceremony on 2 September, the former HZB trainee Katrin Kerrmann was honoured by the chancellor for the early completion of her course as a mechatronics engineer and her excellent grades. And especially in view of the fact that young women are still sorely underrepresented in the skilled

technical trades – in the ABB training centre, the contingent of women constitutes only 10 percent. “Initially, I had a very healthy respect for the training course but also reservations as to whether I would be able to prevail in this almost exclusively male domain. But now I'm really happy that I took the plunge,” Katrin Kerrman said within the scope of a report featured in the German ARD television news on her experiences during training. During the so-called vocational sandwich course, in which HZB has participated as a partner since 2005, over 500 trainees hailing from 120 companies are trained and qualified in the ABB training centre. The HZB partici-

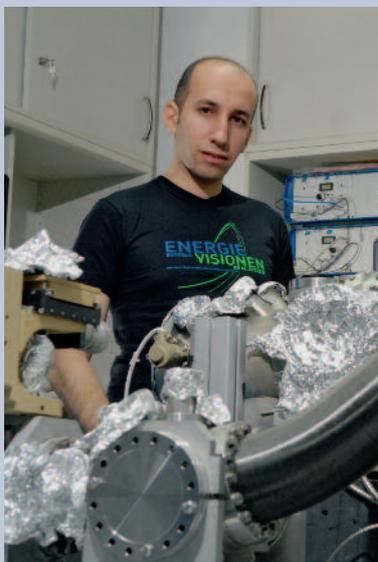


pates with its two training courses for mechatronics engineers and electronics engineers for industrial engineering, which are both given by the long-standing trainer Armin Dellermann. The chancellor gave the young trainees encouragement for their future by saying: “You are now much more sought after than was the case perhaps 10 or 20 years ago”.

## FIRST ERC STARTING GRANT FOR HZB

THE **EUROPEAN RESEARCH COUNCIL (ERC)** IS SUPPORTING A PROJECT BY PROF. DR. EMAD FLEAR AZIZ, SCIENTIST AT HZB AND THE FU BERLIN, TO THE TUNE OF 1.5 MILLION EUROS.

With their so-called starting grants, the European Research Council supports aspiring researchers who form their own research team or who want to carry out independent research in Europe. It addresses all those who verifiably bring with them the potential for heading up such research projects. Prof. Dr. Emad Flear Aziz received the accolade in September 2011. It permits him to continue his research into functional materials over the next five years. Over and above this, the Helmholtz Association is sponsoring Aziz' research work with a further 250,000 euros. His research project is called "Structure and Dynamics of Porphyrin-



Based Materials in Solutions vs. Interfaces". The objective of this work is to combine the technology of short-pulse lasers and X-ray light sources to permit investigating the dynamics of biochemical samples in solution. In the process, a short laser pulse is used to trigger chemical or biological reactions. The X-ray pulse that follows hard on its heels permits an insight into the structural changes on the molecular and atomic levels that occur during this process. Emad Aziz can now use this grant to build up an interdisciplinary network for this type of research, which is incidentally expected to last longer than the period of sponsorship.

### IMPORTANT APPOINTMENTS

**Prof. Dr. Susan Schorr**, head of the Crystallography Department at HZB, received a joint professorship from the FU Berlin and HZB in March 2011. She lectures at the FU Berlin's Faculty of Geosciences.

**Prof. Dr. Thomas Hannappel**, formerly provisional head of the HZB department "Materials for Photovoltaics", received an endowed chair for photovoltaics at the Ilmenau University of Technology in August 2011. This endowed professorship goes hand in hand with the scientific management of the solar centre in the CiS Research Institute for Microsensor Systems and Photovoltaics in Erfurt.

**Prof. Dr. Joachim Dzubiella** was offered a joint professorship for "the theory and simulation of multi-particle systems" at the Department of Physics of the Humboldt University in Berlin. The chair is linked to management of an independent work group at HZB.

**Prof. Dr. Alexei Erko** has accepted an honorary professorship at the Freie Universität Berlin (FUB).

**Prof. Dr. Marcus Bär** has accepted a junior professorship in the Faculty of Photovoltaics at the Brandenburg University of Technology Cottbus.

### IN BRIEF

On 19 June 2011, Professor Dr. Christian-Herbert Fischer and his team were selected as one of the four **German High-Tech Champions 2011** for the ion layer gas reaction process (ILGAR) patented by him. This competition, sponsored by the Fraunhofer Society, is intended to promote Germany as a research location abroad. The prize of 10,000 euros made it possible for Fischer and his team to demonstrate their process to produce thin-film solar cells to American companies in Boston.

On 24 June 2011, Prof. Dr. Emad Flear Aziz received the **Karl-Scheel Prize 2011** awarded by the German Physical Society (DPG). With this prize, the society paid tribute to "his excellent work on the structure and dynamics of functional materials in solution". The research group headed up by Emad Aziz was able to find out on a microscopic scale exactly how dissolved biochemical materials exert their function in their natural environment.

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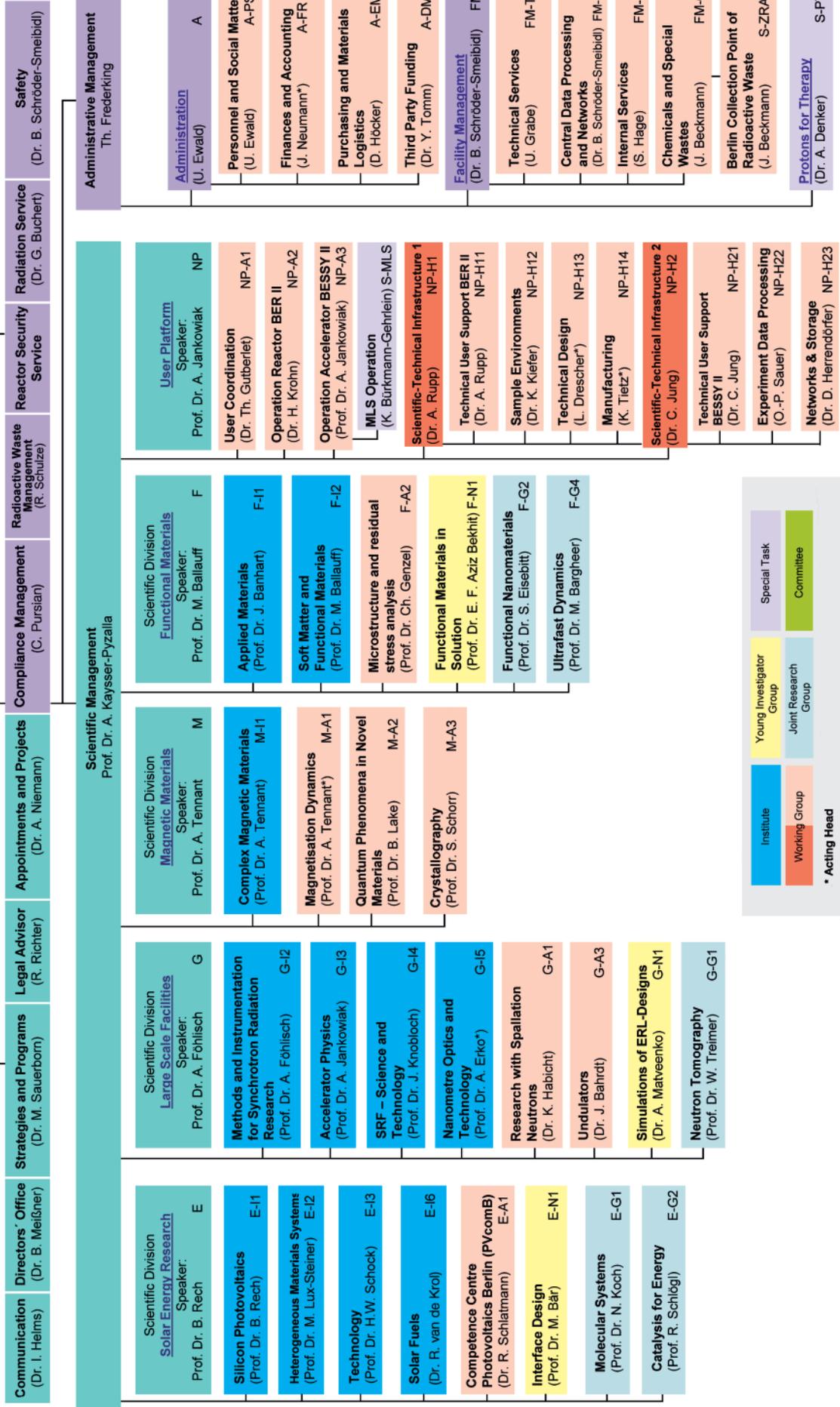
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### Site map

The Lise-Meitner-Campus with the research neutron source BER II is located at the HZB Berlin-Wannsee site, whereas the Wilhelm-Conrad-Röntgen-Campus with the electron storage ring BESSY II is located at the HZB Berlin-Adlershof site.



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