HZB Helmholtz Zentrum Berlin

Soft Matter and Functional Materials

User Facilities – Scientific Activities – Future Perspectives

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and
Functional Materials

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User Facilities - Scientific Activities - Future Perspectives

Edited by Dr. Nikoline Hansen & Dr. Martin Hoffmann

Research on Soft Matter and Functional Materials at the Helmholtz-Zentrum Berlin für Materialien und Energie

The Institute of Soft Matter and Functional Materials

Soft Matter Science is located at the interface between physics, chemistry, and biology, where novel and fascinating research areas are emerging and interdisciplinary approaches are required. Systems belonging to the field of soft matter range from biological macromolecules and their function in life science to industrial colloids that are produced in millions of tons. Investigations of the structure and dynamics of these systems provide a major challenge inasmuch as their typical sizes are located between the atomistic scale as e.g. in the case of proteins and the macroscopic scale going up to dimensions of a cell. The dynamic range is equally impressive since it goes from nanoseconds to hours. Scattering methods are uniquely suited to analyze soft matter systems in a comprehensive way. Thus, methods as e.g. small-angle neutron or X-ray scattering have become indispensable tools in the field of soft matter science.

The Institute of Soft Matter and Functional Materials (F-I2) at the Helmholtz-Zentrum Berlin für Materialien und Energie (HZB), founded in 2009, is devoted to the basic understanding and possible applications of colloidal and nanoscopic systems, of protein structure and function, and of complete cellular compartments. A main focus of our institute is the combination of beamlines with dedicated user laboratories in which complementary methods such as light scattering and electron microscopy are provided for our users. Thus, difficult and sensitive samples can be prepared in our laboratories and analyzed directly at our beamlines. Serving a broad community of scientists from molecular biologists to industrial chemists we provide and develop

- top-class, dedicated beamlines
- complex experimental set-ups (neutrons, photons & more)
- dedicated laboratory infrastructure
- theoretical support (analytical modeling and computer simulations)

Based on our in-house research we allocate excellent beamlines for our users together with an advanced infra-structure. We provide all necessary complementary methods comprising both experimental facilities (user labs, advanced experimental set-up, etc) and offer additional support in theory (MD-simulations). An interdisciplinary team of scientists, PhD-students and technicians from physics, chemistry and biology is working on these topics and closely interacting with our users. In this way our institute provides an inspiring scientific environment for our 15 PhD students.

The development of new beamlines and beamline components is geared by in-house research that is devoted to the following topics:

- Structure and dynamics of proteins
- Analysis of soft- and bio-interfaces as well as thin films
- Synthesis and analysis of the structure and dynamics of colloidal suspensions
- Structural analysis of soft matter by a combination of small-angle scattering and microscopy including X-ray microscopy

Special emphasis is focused on a close collaboration with university groups, particularly from the Berlin area. This collaboration has led to the inauguration of the ***Joint Berlin MX Laboratory*** where research institutions from Berlin (HZB, Freie Universität Berlin (FU), Humboldt-Universität zu Berlin (HU), Max-Delbrück-Zentrum für Molekulare Medizin, Leibniz-Institut für Molekulare Pharmakologie) work together in the field of macromolecular crystallography. Three MX-beamlines located at the HZB are managed by this group. In many respects this joint lab has become the model for successful collaboration of a Helmholtz Zentrum with universities.

In the ***Joint Laboratory for Structural Research*** (JLSR) the HZB, the HU, and the Institut für Kristallzüchtung closely collaborate in the field of structural analysis of soft and hard matter, in particular using microscopic methods. In this lab the HZB and the Institute of Physics of the HU run a high-resolution electron microscope and a cryogenic transmission electron microscope which is fully operative since October 2011.

Moreover, F-I2 has participated in the preparation of four applications of Berlin universities in the ***excellence program*** of the ***Deutsche Forschungsgemeinschaft (DFG).*** We take part in two projects of the newly founded Sonderforschungsbereich HIOS led by the Institute of Physics of the HU. For the time being, we are working in the ***DFG Schwerpunktprogramm*** "Intelligente Hydrogele". Together with international partners we acquired two research grants in the **FP7** of the EU, one on microfluidics and catalysis and another one on the development of the in situ diffraction system in the Macromolecular Crystallography Group. We are also partners in a grant for the development of nanofocusing optics for X-rays with the X-ray Microscopy Group. Furthermore, there are several cooperations with international laboratories and industrial partners.

An important point in the research strategy of F-I2 is the close collaboration with other Helmholtz partners and a close linkage to the Helmholtz Portfolio. Together with the Helmholtz-Zentrum Geesthacht (HZG) we applied successfully for a ***Helmholtz Virtual Institute*** devoted to **Multifunctional Materials for Biomedicine**. Moreover, we take part in the ***Helmholtz Portfolio*** subject "***Technology and Medicine***".

This report gives an overview of the facilities, workshops, statistical information and the inhouse research of the Institute Soft Matter at the HZB. It covers the time span from July 2009 up to January 2012. The main lines of research will be presented together with recent scientific results. Moreover, the CVs of the leading scientists of the institute will be given.

Organization of the Institute

The Institute is subdivided into eight groups and one junior research group:

- Macromolecular Crystallography (Dr. Uwe Müller, Dr. Manfred S. Weiss)
- Biophysics (Dr. Thomas Hauß)
- Colloid Physics (Dr. Günter Goerigk, Dr. Daniel Clemens)
- Interfaces (Dr. Roland Steitz)
- Colloid Chemistry (Dr. Yan Lu)
- X-ray Microscopy (Priv. Doz. Dr. Gerd Schneider)
- Soft Matter Theory (Prof. Dr. Joachim Dzubiella)
- Time-of-Flight-Spectroscopy (Dr. Margarita Russina)
- Junior Research Group Polymer Physics (Dr. Sebastian Seiffert)

Dr. Martin Hoffmann is in charge of industry relations. The Institute is firmly rooted in the scientific environment of Berlin: Dr. J. Dzubiella is W2-S Professor at the HU, Dr. G. Schneider is Privatdozent at the same university (both Dept. Physics), Dr. S. Seiffert has the position of a junior researcher at the FU, Institute of Organic Chemistry, Dr. Yan Lu is teaching at HU, Dr. M. Weiss and Dr. U. Mueller are both teaching with a “Lehrauftrag“ at the FU, Institute of Chemistry and Biochemistry, Dr. M. Russina is teaching in the department of physics of the University of Potsdam and Dr. M. Ballauff is Professor (W3-S) at the Dept. Physics of the Humboldt Universität zu Berlin. In addition to this, Dr. G. Goerigk has a “Lehrauftrag” at the University of Paderborn.

At present (March 2012) the Institute has 59 coworkers that include
12 permanent scientists
22 postdocs
15 PhD-students
5 technicians

The output of the Institute has been 52 publications in refereed journals in 2009, 69 in 2010 and 74 in 2011. This includes 26 papers in journals with an impact factor above 7, 105 in journals with impact between 3 and 7, and 64 in journal below 3.

Beamlines and Laboratories of the Institute

The institute runs **neutron beamlines** on the Wannsee-site as well as **X-ray beamlines at the synchrotron** in Adlershof. The following tables give a survey of the beamlines and the labs of F-I2 together with the group running the facility:

Beamline	*Instrument*	*Group*
V1	Membrane diffractometer	Biophysics
V3	Time-of-Flight Spectrometer (NEAT)	Time-of-Flight-Spectroscopy
V6	Reflectometer	Interfaces
V16	SANS	Colloid Physics
V18	BioRef	Interfaces
BL 14.1	MX-beamline	Macromol. Crystallography
BL 14.2	MX-beamline	Macromol. Crystallography
BL 14.3	MX-beamline	Macromol. Crystallography
U41	X-ray microscope	X-ray microscopy
ASAXS	Small-angle X-ray	Colloid Physics
Electron beam writer	Electron beam writer	X-ray microscopy

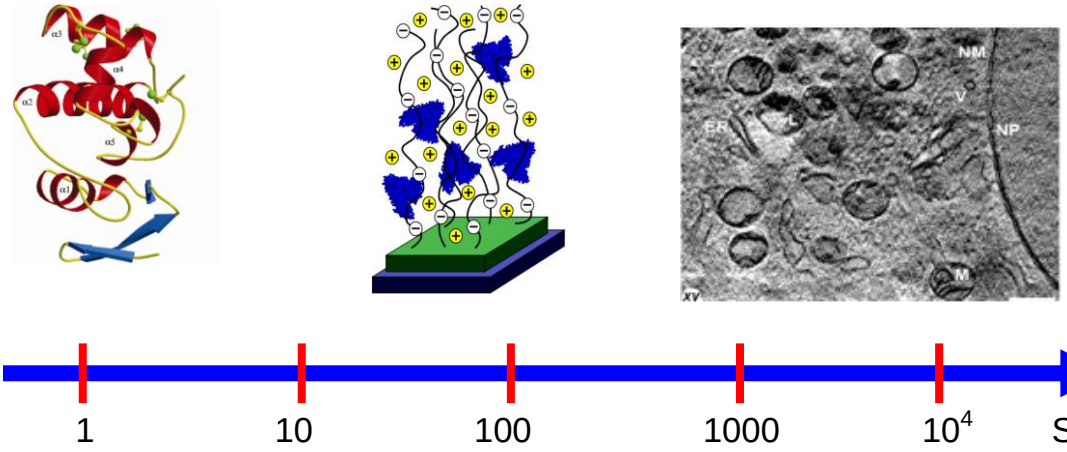
The ASAXS-beamline is run in cooperation with F-A2.

Laboratories	*Group*
BioLab	Macromol. Crystallography/Biophysics
Colloid Lab	Colloid Physics
Chemistry Lab	Colloid Chemistry
Nano Lab for Optical Elements	X-ray Microscopy
Laboratory for Microfluidics	Junior group Polymer Physics
Joint Berlin MX-Laboratory	Macromolecular Crystallography together with partners from the Berlin area
Joint Laboratory for Structural Research	Colloid Physics together with HU-Berlin

Soft Matter and Functional Materials: Research and Recent Highlights

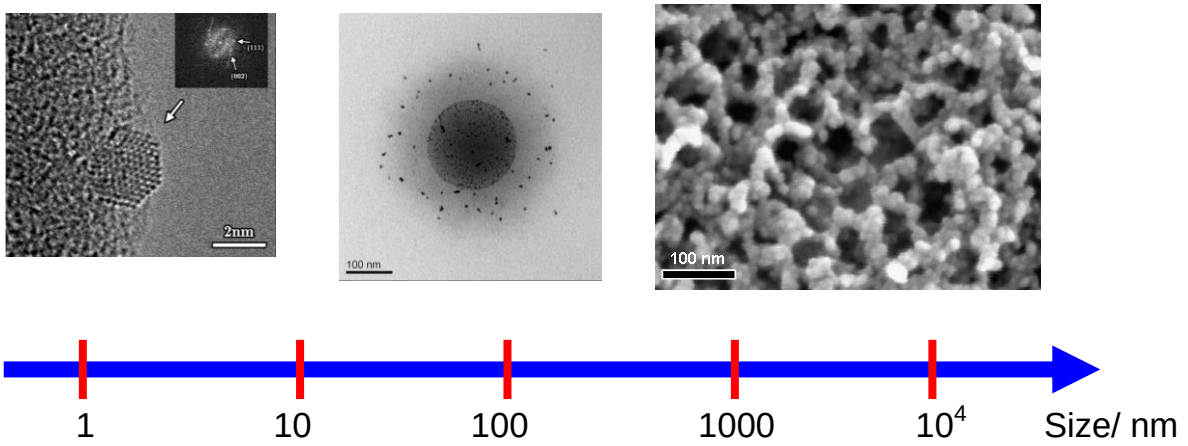
The research of the institute is directed towards the analysis of soft matter throughout all the pertinent length scales as illustrated below. In particular, we study proteins and cellular components in general by starting on the atomistic level. Here a major effort of the Institute is devoted towards protein crystallography. The interaction of proteins with soft polymeric

Proteins **Bio-interfaces** **Cells**



surfaces is a problem of high technical relevance. We investigate this interaction on planar surfaces as well as on nanoparticles by a wide range of methods as e.g. neutron reflection, SAXS, and calorimetry. Finally, entire cells can be studied by cryo-X-ray microscopy that allows us to visualize the different compartments of the cells without staining or microtome sectioning.

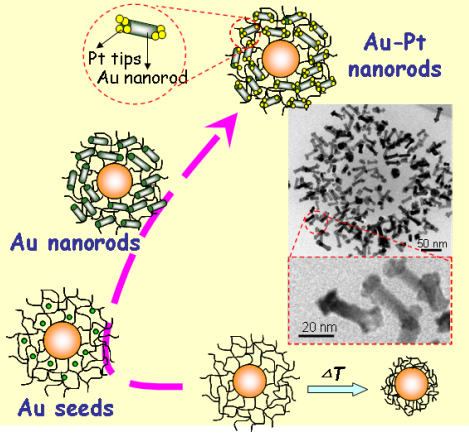
Nanoparticles **Composites** **Mesoporous materials**



Polymeric colloidal particles are studied in a similar way. Here we start from the synthesis of these systems including hybrids of inorganic nanoparticles with polymer colloids (see above). The various beamlines and labs provide an ideal place for analyzing these systems since all methods in the reciprocal and in direct space are available. The groups of the Institute work closely together in collaboration with many research institutions in the Berlin/Brandenburg area. In all cases we look into possible applications, again preferably together with strong partners. Therefore we also strengthen our collaborations with industrial partners in order to exploit possible applications as quickly as possible.

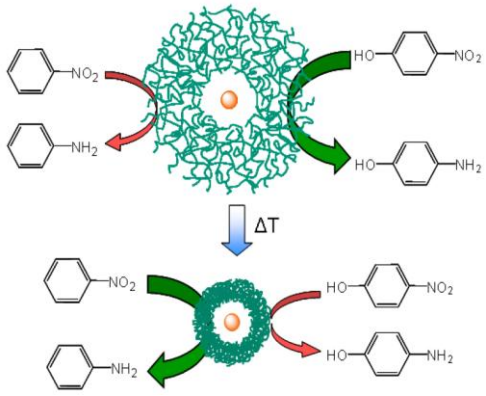
Polymers and nanocomposites

In-situ Growth of Catalytic Active Au-Pt Bimetallic Nanorods in Thermo-Responsive Core-Shell Microgels



Bimetallic Au-Pt nanorods (NRs) can be grown in situ into thermosensitive core-shell microgel particles by a novel two-step approach. In the first step, Au NRs with an average width of 6.6±0.3 nm and length of 34.5±5.2 nm (aspect ratio 5.2±0.6) were homogeneously embedded into the shell of PNIPA networks. The volume transition of the microgel network leads to a strong red shift of the longitudinal plasmon band of the Au NRs. In the second step, platinum was preferentially deposited onto the tips of Au NRs to form dumbbell-shaped bimetallic nanoparticles. The novel synthesis forms bimetallic Au-Pt NRs immobilized in microgels without impeding their colloidal stability. Quantitative analysis of the catalytic activity for the reduction of 4-nitrophenol indicates that bimetallic Au-Pt NRs show highly enhanced catalytic activity, which is due to the synergistic effect of bimetallic nanoparticles. The catalytic activity of immobilized Au-Pt NRs can be modulated by the volume transition of thermosensitive microgels. This demonstrates that core-shell microgels are capable of serving as “smart nanoreactors” for the catalytic active bimetallic nanoparticles with controlled morphology and high colloidal stability.[1]

Thermosensitive Au-PNIPA Yolk-Shell Nanoparticles with Tunable Selectivity for Catalysis



Recently, at our institute an inorganic-organic hybrid yolk-shell (YS) nanostructure was developed that contains metallic Au nanoparticle as the core and a thermosensitive microgel PNIPA as the shell. YS structures have the clear advantages that individual metal nanoparticles are enclosed in a compartment, and the embedded gold nanoparticle has a free surface which is not blocked by any surface group or polymer compared to conventional core-shell structures. Moreover, the permeability of the shell may be tuned to a certain extend This hybrid is shown to be an effective catalyst for the reduction of nitrophenol and nitrobenzene in aqueous solution. Temperature can be used as a trigger to enhance the selectivity of the catalysis for a given substrate: 4-NP reacts much faster at low temperature while the reduction of NB is preferred at higher temperature. This selectivity is even enhanced in mixtures of 4-NP and NB. Hence, yolk-shell systems have a great potential to tailor the catalytic activity and selectivity of metal nanoparticles toward a given reaction.[2]

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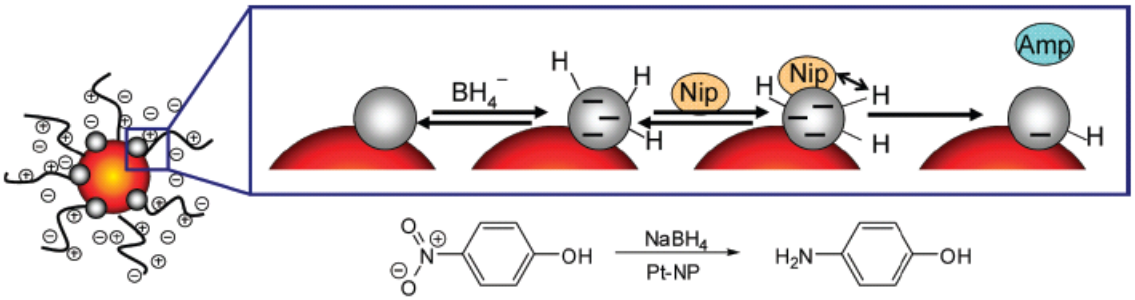
[1] Y. Lu, J. Yuan, F. Polzer, M. Drechsler, J. Preussner, *ACS Nano* **2010**, 4, 7078-7086.
[2] S. Wu, J. Dzubiella, J. Kaiser, M. Drechsler, X. Guo, M. Ballauff, Y. Lu, *Angew. Chem. Intern. Ed*. **2012**, DOI:10.1002/anie.201106515.

Kinetic Analysis of Catalytic Reduction of 4-Nitrophenol by Metallic Nanoparticles Immobilized in Spherical Polyelectrolyte Brushes

The nanoparticles are embedded in spherical polyelectrolyte brushes, which consist of a polystyrene core onto which a dense layer of cationic polyelectrolyte brushes are grafted. The average size of the nanoparticles is approximately 2 nm. The kinetic data obtained by monitoring the reduction of 4-nitrophenol by UV/vis-spectroscopy could be explained in terms of the Langmuir-Hinshelwood model: The borohydride ions transfer a surface-hydrogen species in a reversible manner to the surface. Concomitantly, 4-nitrophenol is adsorbed and the rate-determining step consists of the reduction of nitrophenol by the surface-hydrogen species. The apparent reaction rate can therefore be related to the total surface S of the nanoparticles, to the kinetic constant k related to the rate-determining step, and to the adsorption constants *K*(Nip) and *K*(BH_4) of nitrophenol and of borohydride, respectively. In all cases, an induction time t(0) was observed of the order of minutes. The reciprocal induction time can be treated as a reaction rate that is directly related to the kinetics of the surface reaction because there is a linear relation between 1/(kt(0)) and the concentration of nitrophenol in the solution. All data obtained for t(0) so far and a comparison with data from literature indicate that the induction time is related to a slow surface reconstruction of the nanoparticles, the rate of which is directly related to the surface reaction.[3]



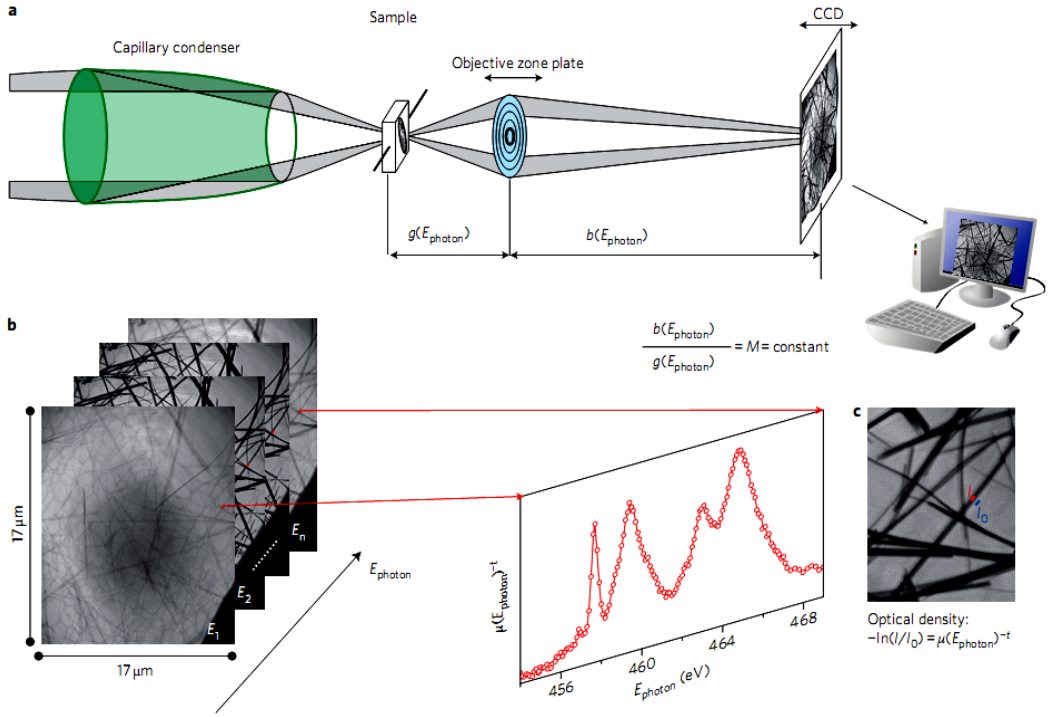
Mechanistic model (Langmuir-Hinshelwood mechanism) of the reduction of Nip by borohydride in the presence of metallic nanoparticles (gray spheres). The nanoparticles are bound to spherical polyelectrolyte brush (SPB) particles that consist of a polystyrene core and a shell of cationic polyelectrolyte chains. The catalytic reduction proceeds on the surface of the metal nanoparticles: The nanoparticles react with the borohydride ions to form the metal hydride. Concomitantly, nitrophenol adsorbs onto the metal surface. The adsorption/desorption of both reagents on the surface is fast and can be modeled in terms of a Langmuir isotherm. The rate-determining step is the reduction of the adsorbed Nip to Amp, which desorbs afterwards.[3]

Nanoscale spectroscopy with polarized X-rays by NEXAFS-TXM

Near-edge X-ray absorption spectroscopy (NEXAFS) is an essential analytical tool in material science. Combining NEXAFS with scanning transmission X-ray microscopy (STXM) adds spatial resolution and the possibility to study individual nanostructures. Here, we describe a full-field transmission X-ray microscope (TXM) that generates high-resolution, large-area NEXAFS data with a collection rate two orders of magnitude faster than is possible with STXM. The TXM optical design combines a spectral resolution of $E/\Delta E = 10^4$ with a spatial resolution of 25 nm in a field of view of 15–20 μm and a data acquisition time of approx. 1 s. As an example, we present image stacks and polarization-dependent NEXAFS

[3] S. Wunder, F. Polzer, Y. Lu, Y. Mei, M. Ballauff, *J.Phys. Chem. C* **2010**, *114*, 8814

spectra from individual anisotropic sodium and protonated titanate nanoribbons. Our NEXAFS-TXM technique has the advantage that one image stack visualizes a large number of nanostructures and therefore already contains statistical information. This new high-resolution NEXAFS-TXM technique opens the way to advanced nanoscale science studies.[4]



Workflow for NEXAFS-TXM measurements. **a**, X-ray optical set-up of the TXM for NEXAFS studies. Monochromatized radiation from the undulator is focused by a reflective capillary condenser into the object field. A zone plate objective forms a magnified image. By choosing regions of interest (ROIs), the optical density can be calculated for each ROI using the sample-free region in its vicinity. As many nanostructures are within the field of view, one photon energy stack contains statistical information. **b**, Stack of (Na,H)TiNR images recorded at different energies. The NEXAFS spectrum is recorded on a ROI (shown in **c**). **c**, Schema showing a ROI for recoding the transmitted photon flux (I) and incident photon flux (I_0).[4]

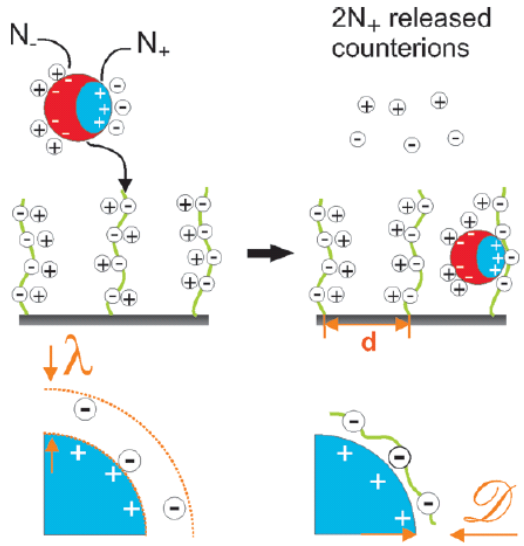
Colloids and microemulsions

Adsorption of beta-Lactoglobulin on Spherical Polyelectrolyte Brushes: Direct Proof of Counterion Release by Isothermal Titration Calorimetry

The thermodynamics and the driving forces of the adsorption of beta-lactoglobulin on spherical polyelectrolyte brushes (SPB) can be investigated by isothermal titration calorimetry (ITC). The SPB consist of a polystyrene core onto which long chains of poly(styrene sulfonate) are grafted. Adsorption isotherms are obtained from measurements by ITC. The analysis by ITC shows clearly that the adsorption process is solely driven by entropy while $\Delta H > 0$. This finding is in accordance with the proposed mechanism of counterion release: Patches of positive charges on the surface of the proteins become multivalent counterions of the polyelectrolyte chains, thereby releasing the counterions of the protein and the polyelectrolyte. A simple statistical-mechanical model fully corroborates the proposed mechanism. The present analysis shows clearly the fundamental importance of

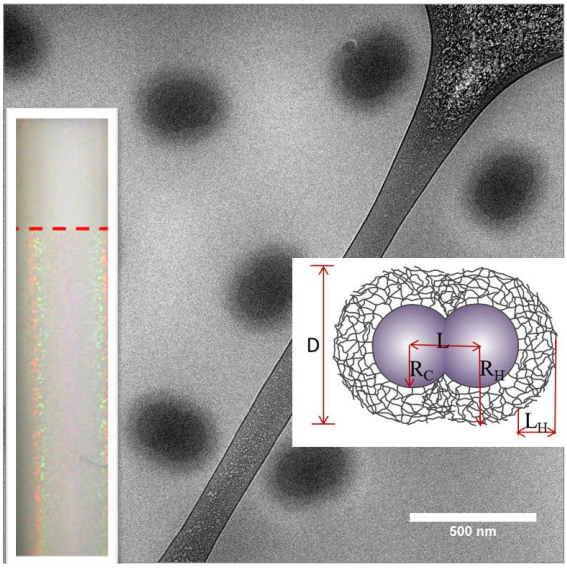
[4] P. Guttmann, C. Bittencourt, S. Rehbein, P. Umek, X. Ke, G. van Tendeloo, C. P. Ewels, G. Schneider, *Nature Photonics* **2012**, *6*, 25.

counterion release for protein adsorption on charged interfaces and charged polymeric layers.[5]



Schematic illustration of the electrostatic model used for the description of the protein interaction with polyelectrolyte brushes. The protein surface carries negatively charged groups. The number N_- of these groups is slightly greater than the number of positive charges N_+ (if pH > p*I*). During the adsorption process the positive patch on the protein surface becomes a N_+-fold counterion of the polyelectrolyte chains in the brush layer. This releases N_+ negative counterions of this positive patch together with N_+ positive counterions of the brush layer. The Gouy-Chapman length of the dissolved protein is λ. This is depicted in the bottom of the left panel. The thickness of the adsorbed polyelectrolyte layer on the protein surface, *D*, is illustrated in the bottom of the right panel.[5]

Synthesis and Characterization of Monodisperse Thermosensitive Dumbbell-Shaped Microgels

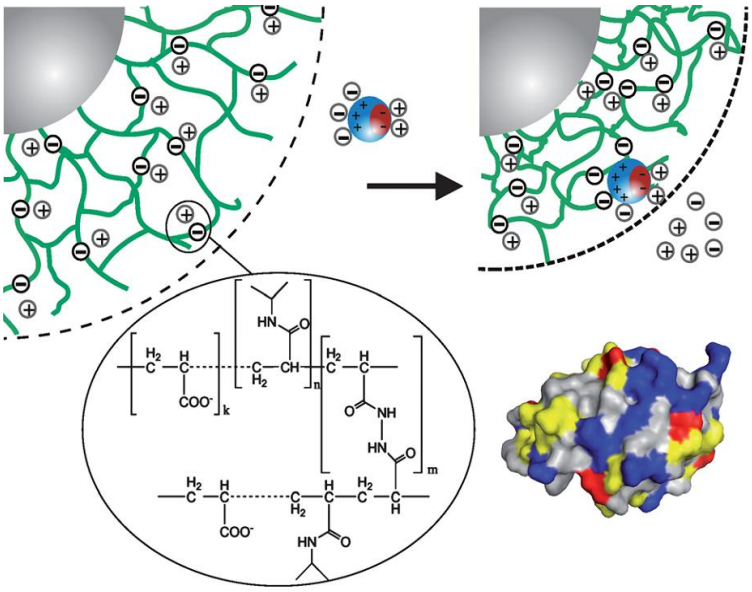


The synthesis and characterization of nearly monodisperse thermosensitive dumbbell-shaped core-shell microgels was recently achieved. The preparation of these microgels was carried out by attaching a thermosensitive shell consisting of a poly(*N*-isopropylacrylamide) network to dumbbell-shaped polystyrene-core particles. The morphology of the microgels was investigated through cryogenic transmission electron microscopy and depolarized dynamic light scattering. The effective volume fraction ϕ_{eff} and aspect ratio *L** (ratio of center to center distance *L* of the spheres to the diameter of the dumbbells *D, L*=L/D*) could be adjusted from *L** approx. 0.22 to 0.26 ± 0.01 through the swelling of the thermosensitive shell within a temperature range from 5 °C to 25 °C. We observe a phase transition of the microgels to an ordered, crystal-like state which is apparent through Bragg-reflections in the visible range. A biphasic gap for $0.50 < \phi_{eff} < 0.56$ is seen, and the comparison with previous computer simulations strongly suggests that the thermosensitive dumbbells form a plastic crystal. These observations were further supported by rheological measurements where the shear melting of the crystal phase is clearly detected.[6]

[5] K. Henzler, B. Haupt, K. Lauterbach, A. Wittemann, O. Borisov, Ballauff, M., *J. Am. Chem. Soc.* **2010**, *132*, 3159-3163

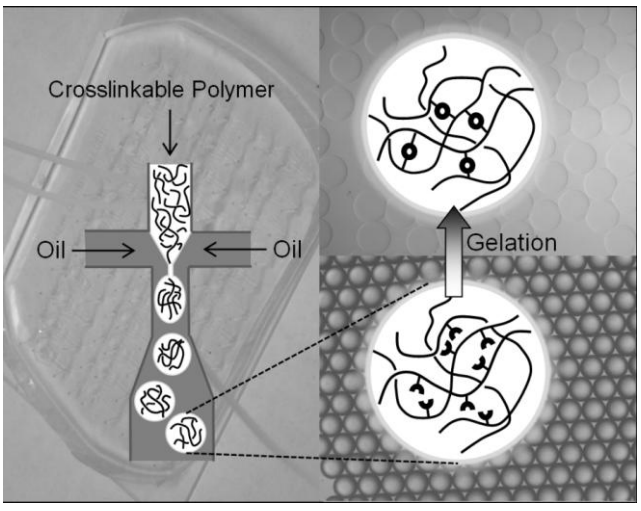
[6] F. Chu, M. Siebenbürger, F. Polzer, C. Stolze, J. Kaiser, M. Hoffmann, N. Heptner, J. Dzubiella, M. Drechsler, Y. Lu, M. Ballauff, submitted to *Macromol. Rapid Commun.*

Core–shell microgels as ‘‘smart’’ carriers for enzymes



A thermodynamic study of the adsorption of lysozyme on a negatively charged core–shell microgel was carried out at pH 7.2. The carrier particles consist of a polystyrene core onto which a charged poly(*N* isopropylacrylamide-co-acrylic acid) network is attached. Isothermal titration calorimetry (ITC) was used to investigate the temperature and salt dependence of lysozyme binding. Our ITC analysis unequivocally shows that the adsorption of lysozyme onto the charged gel is driven by entropy. The addition of salt strongly decreases the binding affinity, indicating significant electrostatic contributions to the adsorption process. However, at high salt concentrations, substantial protein binding with unaltered entropies is still observed pointing to large contributions from hydrophobic interactions. Furthermore, the calorimetric analysis suggests that protonation of lysozyme takes place upon binding. This is directly shown by analysis of the enzymatic activity of adsorbed lysozyme. It was found that the activity is enhanced about ca. 3.5 times, indicating that lysozyme has taken up approximately one proton when entering the gel. The entire set of data demonstrates that core–shell microgels present ‘‘smart’’ colloidal carriers for lysozyme that enhance its activity.[7]

Microfluidic Fabrication of Smart Microgels from Macromolecular Precursor



Stimuli-responsive polymer microgels can be produced with exquisite control using droplet-based microfluidics; however, in existing methods, the droplet templating is strongly coupled to the material synthesis, because droplet solidification usually occurs through rapid polymerization immediately after the microfluidic droplet formation. This circumstance limits independent control of the material properties and the morphology of the resultant microgel particles. To overcome this limitation, we produce sensitive polymer microgels from pre-fabricated precursor polymers. We use microfluidic devices to emulsify semidilute solutions of crosslinkable poly(N-isopropylacrylamide) and solidify the drops via polymer-analogous gelation. This approach separates the polymer synthesis from the particle gelation and allows each to be controlled independently, thus enabling us to form

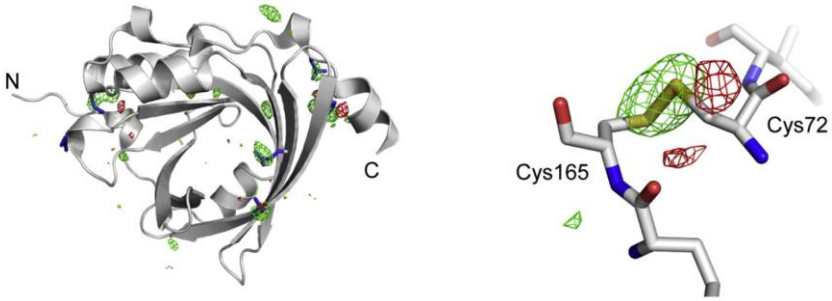
[7] N. Welsch, A. L. Becker, J. Dzubiella, M. Ballauff. *Soft Matter* **2012**, *8*, 1428.

monodisperse, thermoresponsive microgel particles with well-controlled composition and functionality. In addition, the microfluidic templating allows us to form complex particle morphologies such as hollow gel shells, anisotropic microgels, or multi-layered microgel capsules.[8]

Cellular components

Phase determination Using the UV-Light Induced Radiation Damage

After the collection of an X-ray diffraction data-set from a macromolecule crystal the solution of the so-called “crystallographic phase problem” is the major task, which must be resolved. In order to achieve this, a growing number of methods exist, which we are aiming to extend with the further development of the UV-based radiation induced phasing (UVRIP) method. This experimental technique is focused on specific structural changes of cystine-containing protein crystals, which is due to the irradiation of the specimen with highly-intense UV-radiation. The structural changes can be used to work out a single isomorphous replacement (SIR)-like phasing scheme, which can lead to precise experimental phase information and thus to the access to the three dimensional structure (Figure). For this, a native data-set has to be collected before UV-exposure and compared with a second data set collected after the UV-irradiation of the same crystal. At the HZB-MX beamline BL14.1, we have installed all required instruments to carry out such experiments and are providing this to the user community. Within this research project, we aim to develop this method to reduce the existing requirements in terms of minimal data-set resolution and to investigate alternative specific damage sites within a macromolecular crystal.[9]



Specifically damaged disulfide-bridges of the protein α1-acid glycoprotein

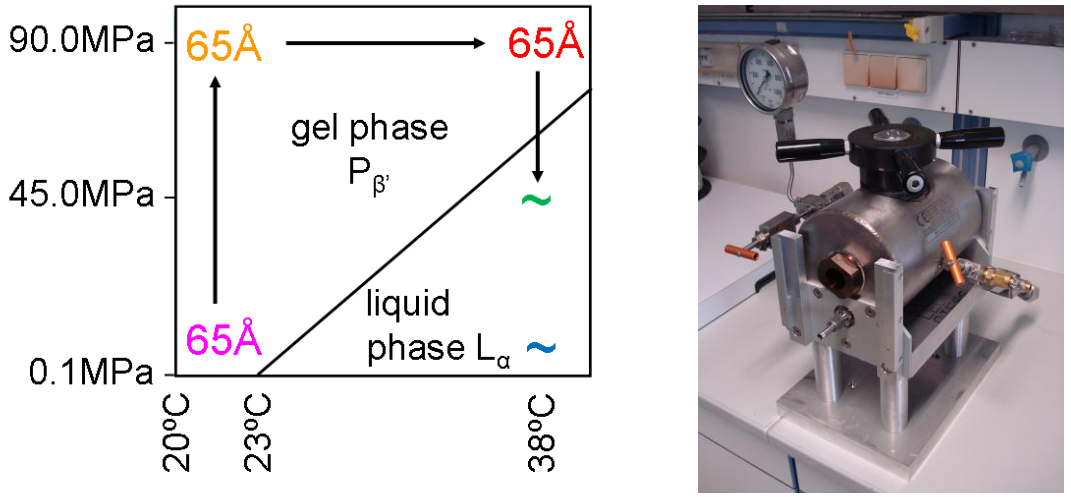
Pressure cell for Investigations of Solid-Liquid Interfaces by Neutron Reflectivity

We describe an apparatus for measuring scattering length density and structure of molecular layers at planar solid–liquid interfaces under high hydrostatic pressure conditions. The device is designed for in situ characterizations utilizing neutron reflectometry in the pressure range 0.1–100 MPa at temperatures between 5 and 60 °C. The pressure cell is constructed such

[8] S. Seiffert, D. A. Weitz, *Polymer* **2010**, 51, 5883.

[9] A. Faust, S. Puehringer, N. Darowski, S. Panjikar, K. Diederichs, U. Müller, M. S. Weiss, *J. Appl. Cryst.* **2010,** *43*, 1230.

that stratified molecular layers on crystalline substrates of silicon, quartz, or sapphire with a surface area of 28 cm^2 can be investigated against noncorrosive liquid phases. The large substrate surface area enables reflectivity to be measured down to 10^{-5} (without background correction) and thus facilitates determination of the scattering length density profile across the interface as a function of applied load. Our current interest is on the stability of oligolamellar lipid coatings on silicon surfaces against aqueous phases as a function of applied hydrostatic pressure and temperature but the device can also be employed to probe the structure of any other solid–liquid interface.[10]



Summary of experimental findings (d-spacings) for the oligolamellar DMPC bilayers film against excess water (D_2O), left. Note that the lipid film irreversibly detaches from support after crossing the phase boundary of the corresponding bulk system at 69 MPa and 38 °C. Photograph of the high pressure cell for neutron reflectometry (right).[10]

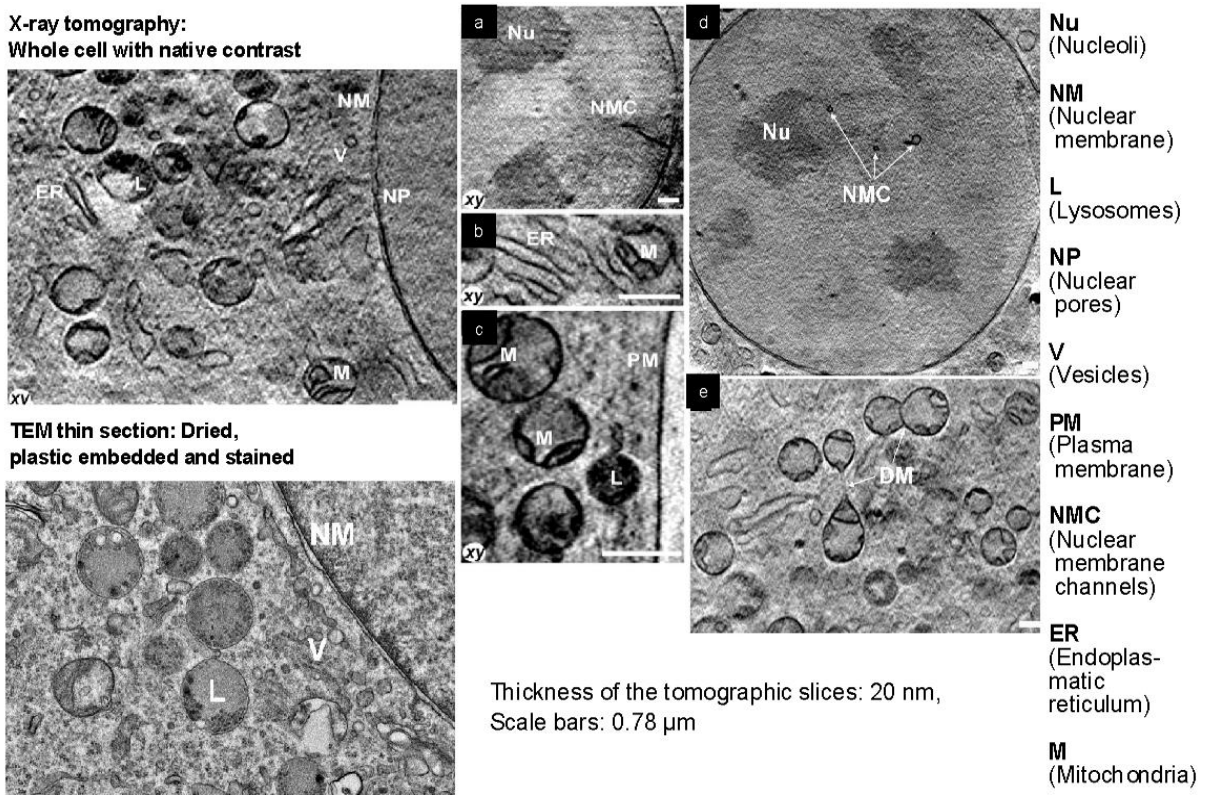
Biomaterials

Three-Dimensional Cellular Ultrastructure Resolved by X-ray Microscopy

We developed an X-ray microscope using partially coherent object illumination instead of previously used quasi-incoherent illumination. The design permitted the incorporation of a cryogenic tilt stage, enabling tomography of frozen-hydrated, intact adherent cells. We obtained three-dimensional reconstructions of mouse adenocarcinoma cells at 36 nm (Rayleigh) resolution, which allowed us to visualize the double nuclear membrane, nuclear pores, nuclear membrane channels, mitochondrial cristae and lysosomal inclusions.[11]

[10] M. Kreuzer, T. Kaltofen, R. Steitz, B. H. Zehnder, R. Dahint, *Rev. Sci. Instr.* **2011**, *82*, 023902-7

[11] Schneider, G.; Guttmann, P; Heim, S; Rehbein, S; Mueller, F; Nagashima, K; Heymann, JB; Muller, WG; McNally, JG : Three-dimensional cellular ultrastructure resolved by X-ray microscopy. Nature Methods 7 **2010**, p. 985.

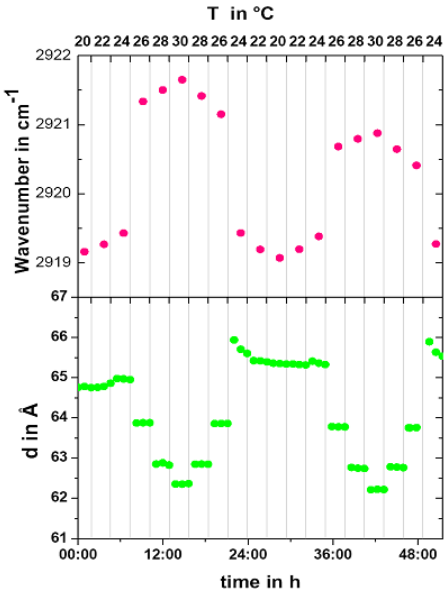


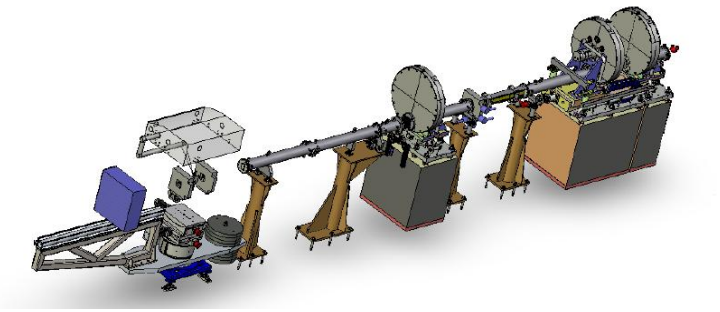
Comparison: Cryo X-ray tomography (upper left) and TEM thin section preparation (lower left). The slices of the X-ray tomograms (a-e) of frozen-hydrated mouse adenocarcinoma cells reveal numerous sub-cellular organelles including dividing mitochondria (DM), vesicles (V), the nuclear membrane (NM), nuclear pores (NP), nucleoli (Nu) and nuclear membrane channels (NMC).

BioRef – a Versatile Time-of-Flight Reflectometer for Soft Matter Applications at Helmholtz-Zentrum Berlin (in Cooperation with Ruprecht-Karls-Universität Heidelberg)

BioRef is a versatile novel time-of-flight (TOF) reflectometer featuring a sample environment for in-situ infrared spectroscopy at the reactor neutron source BER II of the Helmholtz Zentrum Berlin für Materialien und Energie (HZB). After two years of design and construction phase the instrument has recently undergone commissioning and is now available for specular and off-specular neutron reflectivity measurements. BioRef is especially dedicated to the investigation of soft matter systems and studies at the solid/liquid interface. Due to flexible resolution modes and variable addressable wavelength bands that allow for focusing onto a selected scattering vector range, BioRef enables a broad variety of surface and interface investigations and even kinetic studies with sub-second time resolution. The instrumental settings can be tailored to the specific requirements of a wide range of applications. The performance is demonstrated by several reference measurements, and the unique option of in-situ on-board infrared spectroscopy is illustrated by the example of a phase transition study in a lipid multilayer film. [12]

[12] M. Strobl, R. Steitz, M. Kreuzer, M. Rose, H. Herrlich, F. Mezei, M. Grunze, R. Dahint, *Rev. Sci. Instrum.* **2011**, *82*, 055101.

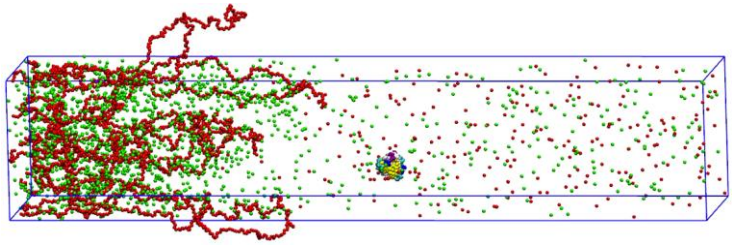




Analysis of lipid bilayers by the new BioRef beamline: d-spacing of an oligolamellar lipid bilayers coating on solid support against an excess water phase and corresponding ATR-FTIR signal of the asymmetric vibrational mode of CH_2 groups of the lipid chains as a function of sample temperature (left) as measured in-situ and combined at BioRef (right).[12]

Proteins and polyelectrolyte brushes

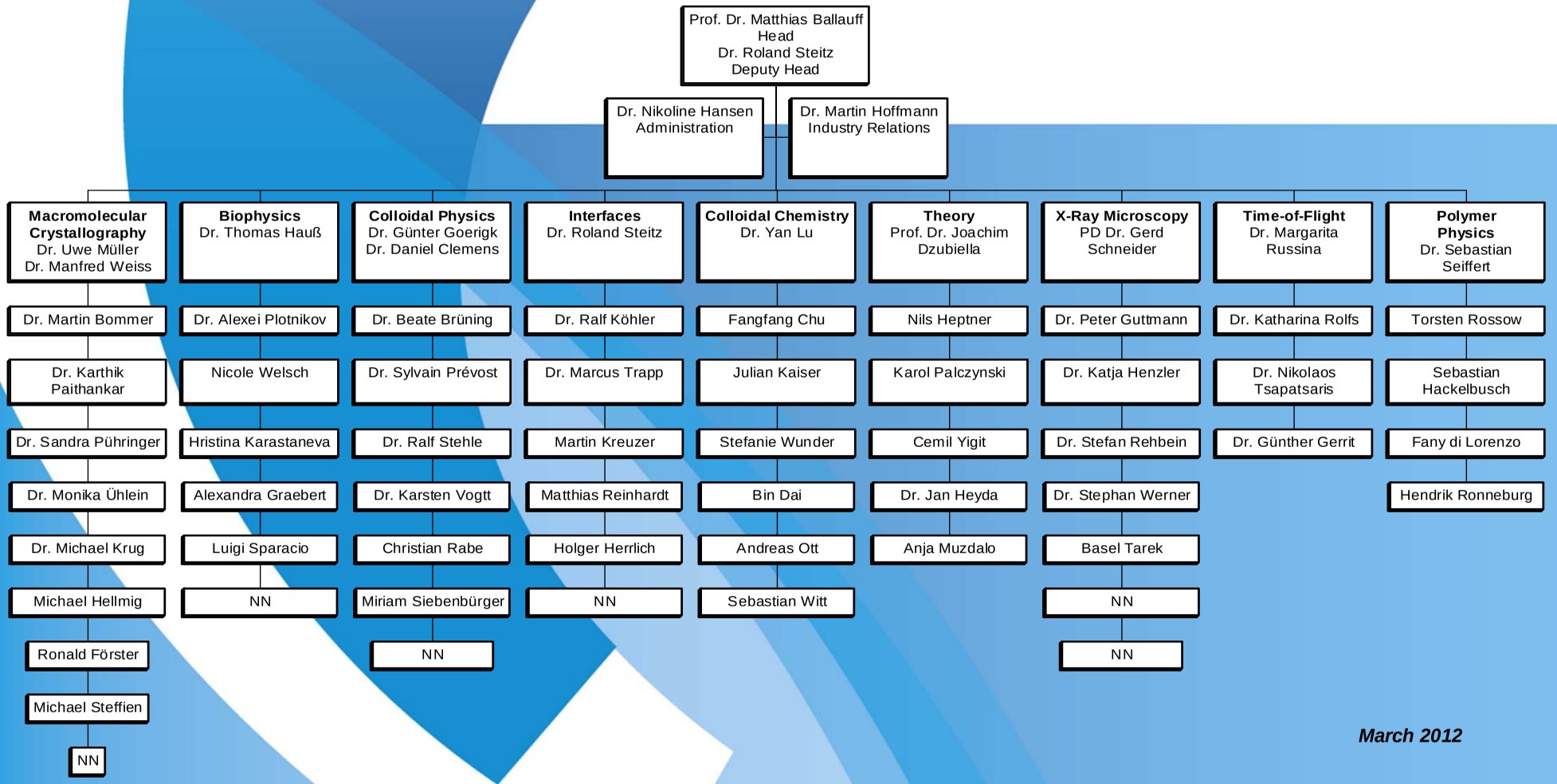
The in-house theory group investigates the physical properties of soft (biological) condensed matter systems using tools of statistical mechanics *and various computer simulation techniques*, such as molecular dynamics, Brownian dynamics (BD), or Monte-Carlo approaches. The scientific topics cover a broad range of systems and include the protein adsorption to soft interfaces or the self-organization of organic materials at inorganic surfaces. These topics are investigated in tight mutual exchange with the experiments at F-I2 and thus directly complement the experimental findings with physical insight, theoretical interpretation, and guidance. Recent highlights are the qualitative theoretical description of diffusion-controlled reactions in the catalysis at yolk-shell nanoparticles,[2] numerical solutions for geometry-hydrodynamics relationships for colloidal dumbbells[6] and the theoretical interpretation of adsorption free energies of proteins at core-shell microgels measured by isothermal titration calometry (ITC).[7] For the latter, the theory group is currently developing a novel, self-consistent fitting framework for the separation of electrostatic and hydrophobic energies incorporating swelling effects of the soft interface; this should have a long-term impact for the interpretation of protein adsorption mechanisms in a variety of soft systems. For microscopic insights we currently perform monomer-resolved computer simulations of polyelectrolye brushes, globular proteins, and specific salt-protein interactions.[13]



BD computer simulation snapshot of lactoglobulin (colored globule in the middle) adsorbing on a planar and charged polyelectrolyte brush (red polymers). Co- and counterions are depicted by small red and green spheres, respectively. The simulation provides microscopic insight into the detailed electrostatic binding mechanisms of the protein on the brush depending on grafting density, poleylectrolyte charge, and salt concentration.

[13] A. H. Crevenna, N. Naredi-Rainer, D. C. Lamb, R. Wedlich-Söldner, J. Dzubiella, *Biophys. J.,* **2012** (accepted**)**

Institute
Soft Matter and Functional Materials



March 2012

Workshops

For an interdisciplinary exchange of experience between scientists in the field of photon and neutron scattering, our institute F-I2 at HZB continuously organized top-class (international) workshops.
The following table summarizes the activities for the years 2010 and 2011.
Recent examples are given subsequently.

Workshop	Organizers	Time
Scientific Kick-off Meeting plus MC-Meeting of the COST Action MP0901 Designing novel materials for nanodevices - from Theory to Practice (NanoTP) *International scientific workshop*	P.Guttmann	18.03.2010 - 19.03.2010
Innovative Instrumentierung zur Untersuchung nanostrukturierter, weicher und biologischer Materie *Workshop of the BMBF-Kompetenzverbund NanoSOFT*	R. Steitz	06.05.2010 - 07.05.2010
Joint Berlin MX Day	U.Müller, M. Weiss, N. Darowski, K. Paithankar, M. Hellmig, S. Pühringer, R. Förster, M. Krug	23.08.2010 - 23.08.2010
XM-Users-Meeting *International scientific workshop*	G. Schneider	03.03.2011
Energy materials research by neutron and synchrotron radiation *International scientific workshop*	M. Russina	08.05.2011 - 11.05.2011
Cryo Soft X-ray Tomography Meeting: Effect of Depth of Focus *International scientific workshop*	G. Schneider	06.06.2011
DGK-Workshop “Diffraction Data Collection Using Synchrotron Radiation” *International scientific workshop*	U. Müller, M. Weiß	03.07.2011 - 08.07.2011
WP-I3 Workshop "Reflectometry for ESS – demands and perspectives" *International scientific workshop*	S. Mattauch (FZJ), J.-F. Moulin (HZG), R. Steitz (HZB)	13.10.2011
ASAXS Workshop *International scientific workshop*	G. Goerigk, A. Höll	24.11.2011 - 25.11.2011
Terahertz Workshop *International scientific workshop*	M. Russina, J. Dzubiella, B. Lake	28.11.2011 - 30.11.2011
Kappa Meeting *International scientific workshop*	U. Müller	28.11.2011 - 29.11.2011
New Methods in Macromolecular Crystallography Using Synchrotron Radiation *International scientific workshop*	U. Müller, M. Weiss	30.11.2011

Workshop "ASAXS in Condensed Matter Science" at Helmholtz Centre Berlin in November 2011

Günter Goerigk[1], Armin Hoell[2], Sylvio Haas[3]
[1]F-I2, Helmholtz-Zentrum Berlin, [2]F-I1, Helmholtz-Zentrum Berlin, [3]Humboldt Universität zu Berlin

In close co-operation between the institutes F-I1 and F-I2 the Helmholtz Centre Berlin hosted the Workshop „ASAXS in Condensed Matter Science" from November 24th to 25th. 45 experts from physics, chemistry, catalyst research and materials science joined the conference at the BESSY campus in Adlershof. Anomalous Small-Angle X-ray Scattering is an element-sensitive analytical method, which combines via Synchrotron Radiation the structural analysis on a length scale between 1 and 100 nano-meters with quantitative information like chemical concentrations and volume fractions of the related nano-scaled phases. The structural and quantitative parameters can be correlated to the macroscopic parameters of the analyzed materials like photo-voltaic properties or catalytic activities of colloidal structures.

In the course of about 20 (invited respectively selected) oral presentations, which were supplemented by a poster session, the disentanglement of complex scientific problems like critical phenomena, kinetics of phase transitions, self-assembling of colloids, transport phenomena and catalytic activity have been addressed. Recent scientific results have been discussed from very different fields like solid state physics, colloidal chemistry, functional materials, catalyst research, fuel-cell applications or medical-pharmaceutic applications. A special nuance of the workshop came from the exchange of experiences between experienced scientist and young academics.
The attendee came from different institutes in Europe among others from the European Synchrotron Radiation Facility (ESRF) in Grenoble (France), from the British light source Diamond in Oxfordshire, from the Hungarian Academy of Science and from the Danish Risoe National Lab. From Germany scientists from Universities, different Helmholtz-Institutes and Institutes of the Max-Planck Gesellschaft attended.

The meeting was organized by G.Goerigk, A.Hoell, S.Haas, C.Ciceron and N.Hansen.

Perspectives in Terahertz Spectroscopy with Neutrons

Margarita Russina[1], Bella Lake[2], Joachim Dzubiella[1] and Arno Hiess (ESS)
[1]F-I2, Helmholtz-Zentrum Berlin; [2]M-A2,Helmholtz-Zentrum Berlin

The unique feature of neutron scattering is that it delivers direct microscopic information in both space and time, while other spectroscopic probes are either local (such as hyperfine field methods) or macro-scopic in the spatial dimension (such as light scattering). Time-of-Flight (TOF) spectroscopy with cold neutrons allows us best to explore phenomena in condensed matter on the terahertz frequency range and on a length scale from 0.5 to 100 Å. This method can be applied to study a broad range of phenomena in complex systems, e. g. quantum phenomena in magnetism, microscopic dynamics of proteins, and fast ionic diffusion in conducting systems. A wave of the new projects and the upgrade of the existing ones caused the fast evolution of modern TOF spectroscopy. One example is the upgrade of the TOF instrument NEAT in HZB with an expected 40-fold intensity increase. Higher data rate and new instrumental capabilities will be offered at the instruments at European Spallation Source (ESS) in Lund.

To explore new scientific opportunities offered by neutron TOF spectroscopy we organized the international workshop “Perspectives in Terahertz Spectroscopy with Neutrons” in collaboration with ESS. The workshop took place on November 29-30, 2011 in Helmholtz Zentrum Berlin. The program included sessions on soft matter, magnetism and material science. A short overview on instrumentation was also presented. The main aim of the event was to address the new application directions.

The workshop received a highly positive response from the community and brought together young and experienced scientists who are using or planning to use neutron terahertz spectroscopy for their research. About 80 participants from 13 countries from all over the world attended the workshop and enjoyed exciting talks on chiral magnetism in multiferroics, in-situ levitation study of the behavior in the metallic melts, neutron scattering investigation of the hydrogen storage materials and ionic liquids. The soft matter session was dedicated to the study of water as a mediator of biological interactions. Talks about news scientific problems on the fields of nanowires and semiconductors were also presented and inspired intense discussions.

The workshop confirmed the high importance of neutron TOF spectroscopy in particular for the exploration of nanostructured materials. It also helped to identify two future directions for TOF instrumentation: applications with small samples and utilization of polarization analysis.

DGK-AK1-Workshop ”X-ray Diffraction Data Collection Using Synchrotron Radiation"

Uwe Müller and Manfred S. Weiss
F-I2 Helmholtz-Zentrum Berlin

As part of the activities of the Working Group 1 ”Biological Structures“ of the German Society of Crystallography (DGK), the workshop ”*X-ray Diffraction Data Collection Using Synchrotron Radiation*” was organized for the third time after 2007 and 2009 and held from July 07-09, 2011 at the BESSY II-storage ring of the Helmholtz-Zentrums Berlin in Berlin-Adlershof. The aim of the workshop was to teach theory and practice of a macromolecular crystallography diffraction experiment using synchrotron radiation from preparatory steps to the experiment itself and the analysis of the acquired data. The 20 participants (master students, PhD students and postdocs) from Germany, Austria, the Czech Republic, Denmark, Finland, Israel, Norway, Poland, Sweden and Switzerland were selected from 68 applicants.

Within the 3-day workshop (http://www.helmholtz-berlin.de/events/bessy-mx-workshop/index_de.html) 10 lectures were presented, in which the scientific basics in synchrotron radiation, crystal preparation, data collection and processing, quality indicators of diffraction data, radiation damage and phase determination were discussed. In addition, the students had the opportunity to carry out an actual diffraction experiment. Five groups of four students each worked on the HZB MX-beamlines on their experiment under the supervision of experienced scientists from the MX-group. At the end of the workshop, the students had the opportunity to present their results to the other participants. The scientific program was completed by a poster session during which the participants presented the projects they are working on in their home institute.

Statistics User Service

List of Instruments run by the Institute Soft Matter and Functional Materials:

Instruments in the BER II Cold Neutron Guide Halls

Instrument		Instrument scientist	Phone +49 30 8062-
V1	Membrane Diffractometer	Thomas Hauß	42071, 42202
V3	Time-of-Flight Spectrometer (NEAT)	Margarita Russina	43159
V6	Reflectometer	Roland Steitz Ralf Köhler	42149, 42806 43077, 42806
V16	Very Small Angle Neutron Scattering (VSANS)	Daniel Clemens Karsten Vogtt Miriam Siebenbürger	42280, 43281 10-812 43022, 43281 43029
V18	Reflectometer for biological applications (BioRef)	Markus Trapp	43233

Due to a shutdown of the reactor in October 2010, a neutron guide hall upgrade and an installation of a new cold source no experiments were carried out since 2010-10-02. Thus the statistics does not include the time semester 2011/I to 2012/I.

Instruments run at BESSY II

Instrument		Instrument scientist	Phone +49 30 8062
BL 14.1	State-of-the-art MX beamline	Uwe Müller Karthik S. Paithankar	14974 15156
BL 14.2	Beamline for de novo structure solution using anomalous phasing methods	Karthik S. Paithankar Sandra Pühringer	15156 14869
BL 14.3	Fixed energy beamline	Manfred S. Weiss	13149
U41 X-ray Microscope	Tomography of cryogenic samples; NEXAFS with nanometer resolution	Peter Guttmann Stephan Werner Stefan Rehbein Gerd Schneider Katja Henzler	14749 13181 13165 13131 43198/13175
ASAXS[1]	Small-angle X-ray Soft Matter	Günter Goerigk	15149
Electron beam writer	VISTEC (type EBPG 5000+ ES)	Stefan Rehbein Stephan Werner Gerd Schneider	13165 13181 13131

[1] Run in close cooperation with F-A2 (Hard Matter)

List of laboratories run by the Institute Soft Matter and Functional Materials:

Chemistry Lab		responsible scientist	Phone +49 30 8062
L 130, L131 LS 224	Chemistry Laboratory Microfluidics	Yan Lu Sebastian Seiffert	43191 42294
V108	Sample preparation neutron hall	Yan Lu	43191

Colloid Lab		responsible scientist	Phone +49 30 8062
LS 117	Colloid Laboratory	Daniel Clemens	42280
LS 217	Rheology	Miriam Siebenbürger	43029

BioLab		responsible scientist	Phone +49 30 8062
WCRC	Crystallization	Manfred Weiss	13149
LS 316, LS 330	Biophysics	Thomas Hauß	42071
LR342, LR340	Gene Laboratory	Thomas Hauß	42071
LR338	Biochemistry	Thomas Hauß	42071

Nano Lab		responsible scientist	Phone +49 30 8062
WCRC	X-ray microscopy optical elements	Gerd Schneider	13131

Joint Berlin MX Laboratory		responsible scientist	Phone +49 30 8062
BESSY II	German center of X-Ray crystallography	Uwe Müller Manfred Weiss	14974 13149

Joint Laboratory for Structural Research		responsible scientist	Phone +49 30
Humboldt University	X-ray microscopy, E-beam lithography	Gerd Schneider Katja Henzler	8062-13131 8062-43198
Humboldt University	Cryo-TEM	Matthias Ballauff Yan Lu	8062-43071 8062-43191
Humboldt University	SAXS, SANS	Guenter Goerigk	8062-15149 2093-7905

Instrument Statistics 2009/I – 2012/I

(preliminary data, 2012, January)

V1: Membrane Diffractometer[a)]

	Number of accepted external proposals	Number of allocated external n-days (short term)	Shifts reques-sted	In-house EF propo-sals	In-house EF n-days	Load factor external (LFE)[b)]
2009/I	5	47	17	2	17	1.67
2009/II	6	52	17	2	16	1.71
2010/I	4	52	19	3	23	1.73
2010/II	4	39	6	1	14	1.81

[a)] Not in service for proposals due to neutron guide hall upgrade / reactor shutdown in 2011/I-2012/I
[b)] *LFE* = number of requested days for experiments / remaining days for regular proposals.

V3: Time-of-Flight Spectrometer (NEAT)

The time-of-flight spectrometer NEAT is undergoing an extensive upgrade until 2013.

V6: Reflectometer[c)]

		Total number of proposals	Number of accepted proposals	Load factor external (*LFE*)
2009/I	Total	20	10	3.59
	Short term extern	13	4	
	EF + LT	7	6	
	Not scheduled		1	
	Conducted		9	
2009/II	Total	20	11	2.46
	Short term extern	13	6	
	EF + LT	7	5	
	Not scheduled		0	
	Conducted		11	
2010/I	Total	18	12	1.75
	Short term extern	10	5	
	EF + LT[d)]	8	7	
	Not scheduled		1	
	Conducted		11	
2010/II	Total	19	7	5.05
	Short term extern	14	3	
	EF + LT	5	4	
	Not scheduled		1	
	Conducted		6	

[c)] Reactor shutdown 2010, Oct 2nd and installation of new cold source. Not in service for proposals.
[d)] EF: in house research; LT: long term

V16: Very Small Angle Neutron Scattering (VSANS)

		Total number of proposals	Number of accepted proposals	Load factor external (*LFE*)
2010/II	Total	8	7	0.55
	Short term extern	4	4	
	EF + LT	4	3	
	Not scheduled		7	
	Conducted		0	

Due to setup,commissioning and shutdown of the reactor (installation of new cold source), V16 was not in service for proposals since 2010/II.

V18: Reflectometer for biological applications (Bio Ref)

Until 2011/I, V18 was not available for public user service (proposals).

BL 14.1, BL 14.2, BL 14.3

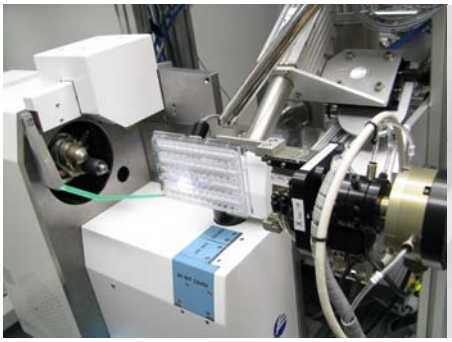
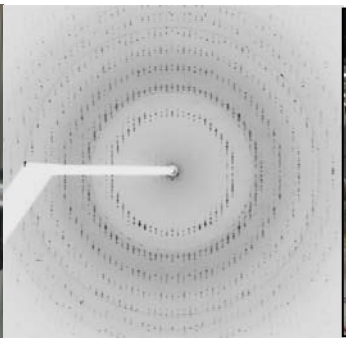
	Number of external Proposals	Number of inhouse proposals	Shifts requested	Shifts granted	Load factor
2009/I	143	11	952	630	1.22
2009/II	153	15	1101	719	1.41
2010/I	160	22	887	578	1.13
2010/II	153	25	1005	62	1.29
2011/I	143	25	885	700	1.13
2011/II	193	24	963	670	1.43
2012/I	170	20	835	612	1.36

U41 X-ray microscope

		2009/I	2009/II	2010/I	2010/II	2011/I	2011/II	2012/I
Number of proposals applied for beamtime:	total	22	30	22	23	18	19	16
	there from inhouse proposals	1	4	2	3	1	1	2
	with approved beamtime	12	17	6	12	12	10	14
	there from inhouse proposals	1	4	1	2	1	1	2
Number of approved shifts for:	In-house users	24	28	22	42	28	24	50
	German users	36	27	22	22	38	3	21
	EU users	13	24	29	24	30	56	51
	Non-EU users	38	24	7	21	20	0	0

Soft Matter and Functional Materials

Beamlines

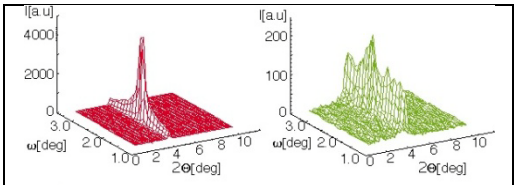




V1

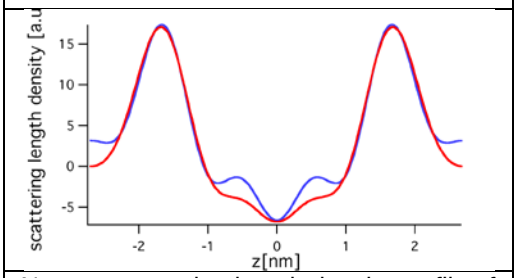
Thomas Hauß

The diffractometer V1 is installed at the curved cold neutron guide NL1A. It is equipped with a high-resolution area detector. The design of the instrument is dedicated for experiments with biological membranes, polymers, microemulsions, micelles and other partly oriented systems. Dedicated sample environment for biological samples and a BioLab for on-site sample preparation is available. An in-situ micro balance and in-situ sorption apparatus is provided by the supply lab for gas adsorption DEGAS.

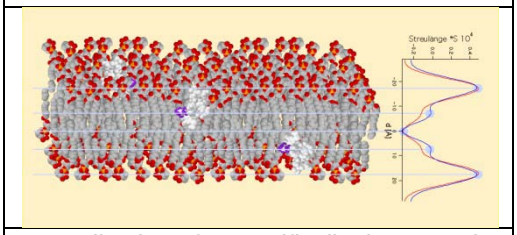
Type	Basic design: 2-axes diffractometer with cradle (± 10°)
Monochromator	pyrolythic graphite (002), vertically focusing
Wavelength	selectable between 0.3-0.6nm (cold neutrons), corresponding to monochromator angles $2\Theta_M$ = 60°-120° (not alterable during experiment)
Angular Range	-10° to 120°
Collimation	γ_0 = 1° at 0.45 nm γ_1: defined by two slit systems
Monochromator-Sample Distance	0.8m - 1.5m (extendable)
Sample-Detector Distance	0.8m - 2.0m
Detector	^{3}He, 19 x 19cm; pixel size 1.5 x 1.5mm^2; height and inclination adjustable



Rocking curves around a Bragg peak of a membrane stack of low and high mozaicity



Neutron scattering length density profile of membranes with and without deuterium label.



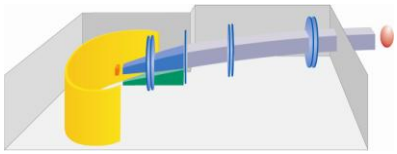
Localization of a specifically deuterated amyloid-b peptide in a lipid membrane with artistic model.

Selected publications

1. Sharifi, M, R Marschall, M Wilhelm, D Wallacher, M Wark: **Detection of homogeneous distribution of functional groups in mesoporous silica by small angle neutron scattering and in situ adsorption of nitrogen or water.** Langmuir 2011, **27**(9):5516-5522.
2. Engelbrecht, T, A Schroeter, T Hauß, RH Neubert: **Lipophilic penetration enhancers and their impact to the bilayer structure of stratum corneum lipid model membranes: Neutron diffraction studies based on the example Oleic Acid.** Biochim Biophys Acta 2011, [Epub ahead of print].
3. Buchsteiner, A, T Hauß, S Dante, NA Dencher: **Alzheimer's disease amyloid-beta peptide analogue alters the ps-dynamics of phospholipid membranes.** Biochim Biophys Acta 2010, **1798**(10):1969-1976.
4. Seelert, H, DN Dani, S Dante, T Hauß, F Krause, E Schäfer, M Frenzel, A Poetsch, S Rexroth, HJ Schwaßmann, T Suhai, J Vonck, NA Dencher: **From protons to OXPHOS supercomplexes and Alzheimer's disease: Structure-dynamics-function relationships of energy-transducing membranes.** Biochim Biophys Acta 2009, **1787**(6):657-671.
5. Mascotto, S, D Wallacher, A Brandt, T Hauß, M Thommes, GA Zickler, SS Funari, A Timmann, BM Smarsly: **Analysis of microporosity in ordered mesoporous hierarchically structured silica by combining physisorption with in situ small-angle scattering (SAXS and SANS).** Langmuir 2009, **25**(21):12670-12681.

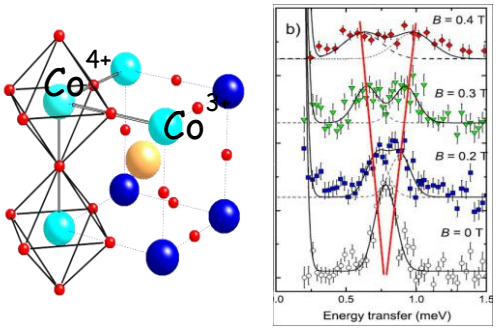
V3

Margarita Russina
Katharina Rolfs
Nikolaos Tsapatsaris

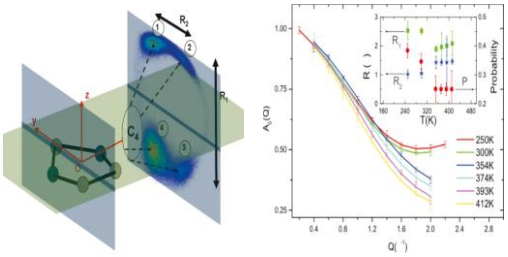


The time-of-flight spectrometer NEAT (V3) is designed for study of the microscopic dynamics in the region of medium to small energy and momentum transfers. NEAT will have an access to very large wavelength range of 2-20 Å with variable energy and angle resolution. NEAT will have an exchangeable focusing guide to adapt best for the experiments with different sample sizes. The instrument design arranges for the application with variable sample environment including high-field magnet of 15 T. Novel position sensitive detectors will cover a large solid angle of 270° and allow for the single crystal applications. NEAT can be used for various scientific problems in magnetism, soft matter and material science. Some examples are: hydrogen storage materials, confinement phenomena in magnetism and soft matter, crystal field splitting, spin dynamics in high-TC-superconductors or dynamics of biological systems and soft matter, including proteins, membranes, hydration water, etc.

Neutron guide	NL2 new (125 x 60 mm)
Exchangeable focussing	trumpet-antitrumpet / parabolic
Chopper speed range	Up to 18000 RPM
Polarization of neutron beam	planned
Incident wavelength range for monochromatic measurements	1Å-20 Å
Detectors	position sensitive detector (270°)
Sample environment	Cryostat 1.5 K – 300 K Cryofurnace 1.5 K – 570 K HT-furnace 300 K – 1270 K Magnetic field 0 T-12 T Humidity chamber Pressure cell



Spin polarons - Magnetic field evolution of INS spectra of $La_{0.998}Sr_{0.002}CoO_3$, *Physical Review Letters* **101,** 247603, (2008).



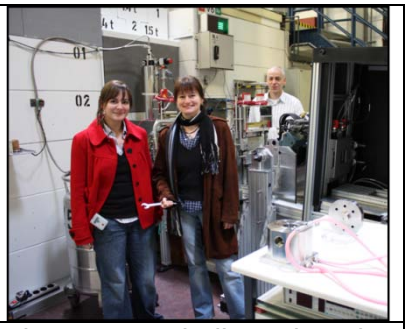
Translational and reorientation microscopic dynamics in imidazolium-based ionic liquid explored by quasielastic neutron scattering and numerical computer simulations *Phys. Chem. Lett.1*,2 **503,** (2010).

Selected Publication:

Z. Izaola, M. Russina, *J. Phys.: Conf. Ser.* **251** 012064, (2010)

V6

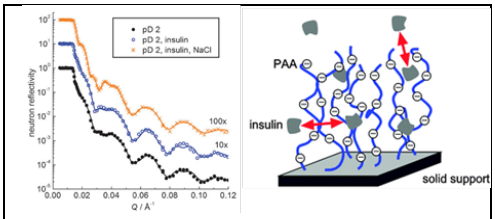
Roland Steitz
Ralf Köhler
Robby Kischnik



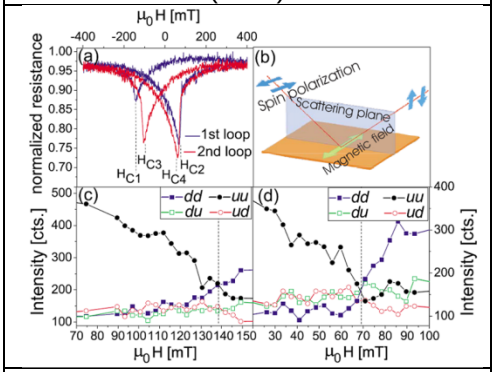
The reflectometer V6 is an angle dispersive fixed wavelength instrument dedicated to the investigation of thin films and surface structures at solid-air and solid-liquid interfaces as well as on free liquid surfaces.
The instrument is optionally equipped with polarization and polarization analysis for studies of magnetic thin films, also in external magnetic fields and at low sample temperature.

Monochromator	pyrolythic graphite (002) mosaicity: Δλ/λ=2%
Wavelength	0.466 nm
Scattering plane	vertical
Polarization of neutron beam	98.5 %
Guide field	Permanent, horizontal
Detectors	^{3}He-detector tubes, position sensitive detector (180 x 180 mm, resolution 1.5 mm)
Q-range [1/Å]	0 - 0.165 (0.127 free liquid surface)
Q-resolution [1/Å]	0.001

Sample environment:
high pressure cell (1000 bar) for solid-liquid interfaces (RKU); heatable cells for liquids, solid-liquid and solid-gas interfaces; Langmuir Blodgett trough;
horizontal magnetic field ≤ 1 T; sample rotation table (360°); closed cycle cryostat (4-300 K);



Probing adsorption and aggregation of insulin at a poly(acrylic acid) brush, Evers, F.; Reichhart, C.; Steitz, R.; Tolan, M.; Czeslik, C.; PCCP **12**, 4375 (2010)



Exchange bias by implantation of O ions into Co thin films, Demeter, J.; Meersschaut, J.; Almeida, F.; Brems, S.; Van Haesendonck, C.; Teichert, A.; Steitz, R.; Temst, K.; Vantomme, A.; Appl. Phys. Lett. 96, 132503 (2010)

Selected publications:

1. Interaction of IAPP and insulin with model interfaces studied using neutron reflectometry, Jeworrek, C.; Hollmann, O.; Steitz, R.; Winter, R.; Czeslik, C.; Biophysical Journal 96, 1115 (2009)
2. Shear Induced Relaxation of Polymer Micelles at the Solid–Liquid Interface, Wolff, M.; Steitz, R.; Gutfreund, P.; Voss, N.; Gerth, S.; Walz, M.; Magerl, A.; Zabel, H.;Langmuir, 24, 11331 (2008)
3. Binding of heavy and light water to polyelectrolyte multilayers, Ivanova, O.; Soltwedel, O.; Gopinadhan, M.; Koehler, R.; Steitz, R.; Helm, C. A.; Macromolecules, 41, 7179 (2008)

V16

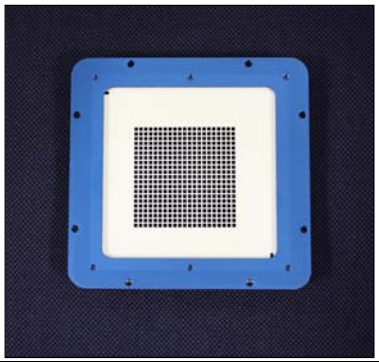
Daniel Clemens
Karsten Vogtt
Marcel Straschewski

The very small-angle scattering instrument V16 (VSANS) serves for the analysis of mesoscopic structures, usually diluted in a buffer liquid. As the method is sensitive to variations in the scattering length density even pores can be investigated. Contrast variation techniques, namely deuteration of sections of the molecules to be investigated are crucial to obtain a maximum of information.

The sample table can be equipped with a thermalized 20 position sample changer, standard cryostats, furnaces or magnets.

Type	Basic design: TOF_SANS
Wavelength Band	0.2 - 1 nm (cold neutrons)
Standard Mode:	
Angular Range	0.2° to 30° (dependent on detector set-up)
Collimation	9 exchangeable guide optics: 1, 2, 4, 6, 8, 10 and 12m
Sample Detector Distance	1.7 m – 11.4 m
Q-range	0.02 nm^{-1} < Q < 16 nm^{-1}
Detector	112 ^{3}He-PSD covering 100 x 100 cm^2; pixel size 84 x 84 mm^2
Low-Q Mode:	
Angular Range	0.05 to 0.75
Collimation	Multi-pinhole optics focusing on detector center
Sample-Detector Distance	11.4 m
Q-Range	0.005 nm^{-1} < Q < 0.4 nm^{-1}
Detector	^{3}He area covering 30 x 30 cm^2; pixel size 2 x 3 mm^2

20-Position sample changer, windows temperature controlled, removable



One of 24 Multi-pinhole diaphragms with 441 pinholes of ~2×2 mm

Selected publication:

1. Mezei, F., Clemens, D., Mokrani, L., *Neutronenoptisches Bauelement für die Neutronen-kleinwinkelstreumesstechnik* - PCT/DE03/02869

V18

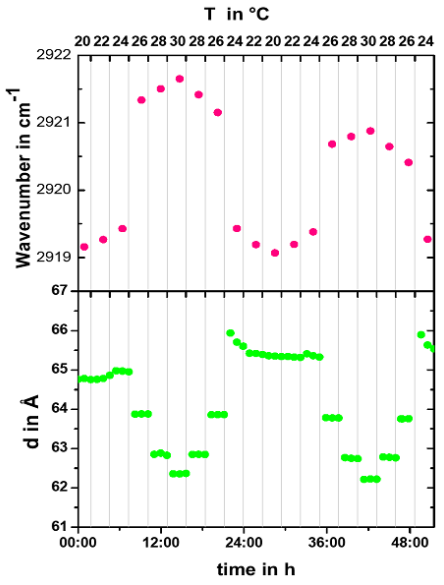
Marcus Trapp
Martin Kreuzer
Werner Graf

The reflectometer V18 BioRef is a versatile time-of-flight instrument dedicated to the investigation of soft matter thin films and interfacial structures at solid-air and solid-liquid interfaces. The chopper system allows for tailoring the resolution of the instrument to the requirements of the specific measurement as well as for kinetic studies.
The instrument is optionally equipped with an infrared spectrometer for simultaneous in-situ measurements in ATR-FTIR geometry.

3-chopper system	Δλ/λ=1% to 12%
Wavelength band	0.25 nm to 0.7 - 1.8 nm
Scattering plane	horizontal
Polarization of neutron beam	Not yet
Guide field	Not yet
Detectors	position sensitive detector (300 x 300 mm, resolution 2 x 3 mm^2)
Q-range [1/Å]	0 - 0.4
Q-resolution [dQ/Q]	1.4 – 20%

Sample environment:

Heatable cells for solid-liquid and solid-gas interfaces;
In-situ ATR-FTIR spectroscopy
Pressure and shear environment under development



Simultaneous neutron reflectivity measurement of the Bragg peak position (green) and infrared spectroscopy measurement of the CH_2 stretching vibration (red) of a lipid multilayer during a temperature scan.

Selected publications:

1. M. Strobl, R. Steitz, M. Kreuzer, A. Nawara, F. Mezei, M. Rose, M. Grunze and R. Dahint, *BioRef – a time-of-flight neutron reflectometer combined with an in-situ infrared spectrometer at the Helmholtz Centre Berlin*, J. of Phys. (conference series) **251** (2010) 012059
2. M. Strobl, R. Steitz, M. Kreuzer, M. Rose, H. Herrlich, F. Mezei, M. Grunze, R. Dahint, ,*BioRef – a versatile time-of-flight reflectometer for soft matter applications at Helmholtz-Zentrum Berlin für Materialien und Energie*, Berlin, Rev. Phys. Instrum. (2011)

BL14.1

Uwe Müller

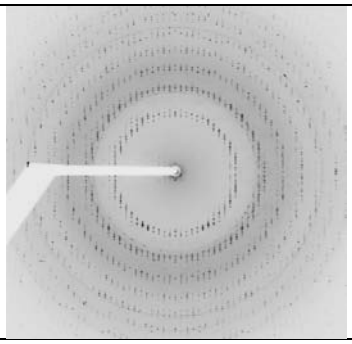
BL14.1 is a state-of-the-art MX beamline and currently the most modern and efficient MX beamline in Germany. It is energy-tunable within the range from 5 keV (2.5 Å) to 16.5 keV (0.75 Å). BL14.1 is equipped with an automatic sample handling robot (CATS). The MD2 microdiffractometer, which is equipped with a mini-kappa goniometer and on-axis sample zoom-microscope, enables the visualization and 3D-centring of crystals at a micrometer scale in the X-ray beam.

Energy range [keV]	5.5 -15.5
Wavelength range[Å]	0.80-2.25 (max. intensity at 0.92)
Max. photon flux at sample [Phot/s/0.1A/0.05% BW]	1.3 x 10^{11} (13 keV)
Energy resolution [eV]	< 2 (9 keV)
Goniometry	Microdiffractometer with Mini-kappa
Sample automation	CATS sample mounting robot (handling of up to 90 SPINE compatible samples)
X-ray detector	Rayonics MX-225
Beam size [μm]	30-100 diameter
Achievable resolution [Å]	0.9
Maximum cell parameter [Å, at 2.0 Å resolution]	400
Exposure time range [sec]	1 - 20

Experimental possibilities:

- High performance *de novo* structure determination by MAD, SAD, SIRAS, MIRAS
- Efficient Screening
- Handling of very small crystals
- RIP, UVRIP
- Element identification using X-ray fluorescence

11 μm crystal within a 50 μm X-ray beam



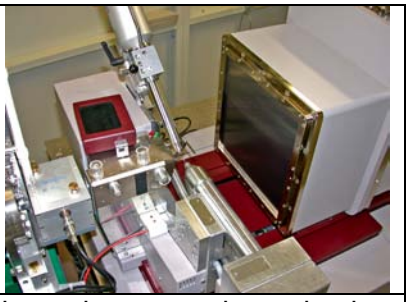
Diffraction image of an aligned protein crystal with reciprocal lattice vector along the X-ray beam

Selected publications:

1. Gao S; Malsburg A; Paeschke S; Behlke J; Haller O; Kochs G; Daumke O, *Structural basis of oligomerization in the stalk region of dynamin-like MxA* 2010, Nature 465, 7297
2. Luckner, S.R., Machutta, C.A., Tonge, P.J., Kisker, C., *Crystal Structures of Mycobacterium Tuberculosis Kasa Show Mode of Action within Cell Wall Biosynthesis and its Inhibition by Thiolactomycin* 2009, Structure 17, pp. 1004
3. Monecke, T., Guttler, T., Neumann, P., Dickmanns, A., Gorlich, D., Ficner, R., *Crystal Structure of the Nuclear Export Receptor CRM1 in Complex with Snurportin1 and RanGTP* 2009, Science 1087

BL14.2

Karthik S. Paithankar
Sandra Pühringer

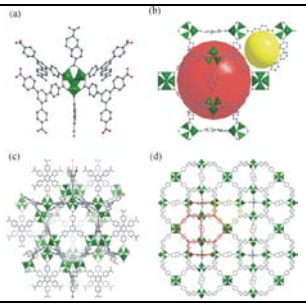


BL14.2 is the workhorse beamline for *de novo* structure solution using anomalous phasing methods such as MAD. The beamline is energy-tunable within the range from 5 keV (2.5 Å) to 16.5 keV (0.75 Å). Bl14.2 is equipped with a mardtb goniometer, which makes it possible to achieve very short detector-to-sample distances of down to 45 mm.

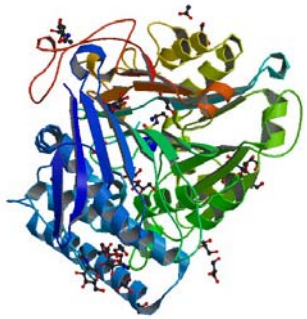
Energy range [keV]	5.5 -15.5
Wavelength range [Å]	0.80-2.25 (max. intensity at 0.92)
Max. photon flux at sample [Phot/s/0.1A/0.05% BW]	1.9 x 10^{11}(13 keV)
Energy resolution [eV]	<2 (9 keV)
Goniometry	MARdtb
Sample automation	No
X-ray detector	Rayonics MX-225
Beam size [μm]	100 x 150

Experimental possibilities:

- High performance *de novo* structure determination by MAD, SAD, SIRAS, MIRAS
- Long wavelength phasing like S-SAD
- Small molecule crystallography application
- Ultra-high resolution data collection
- Element identification using X-ray fluorescence



Crystal structure of mesoporous metal-organic framework (Klein et al. 2009)



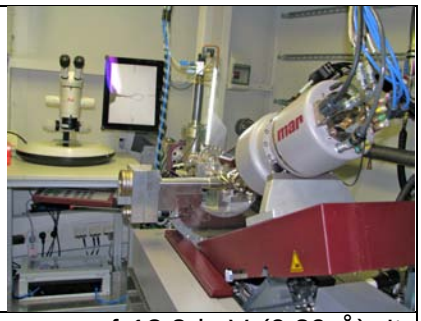
Crystal structure of a 66.3 kDa protein solved by S-SAD (Lakomek et al, 2009)

Selected publications:

1. I Grueninger, D., Treiber, N., Ziegler, M.O.P., Koetter, J.W.A., Schulze, M.-S., Schulz, G.E., *Designed Protein-Protein Association* 2008, Science 319, 206
2. Klein, N,, Senkovska I., Gedrich U, Stoeck U, Henschel A, Mueller U and Stefan Kaskel, *Eine mesoporöse Metall-organische Gerüstverbindung* 2009, Angew. Chem. Int. Ed. Vol. 48, 9954-9957
3. Lakomek, K., Dickmanns, A., Mueller, U., Kollmann, K., Deuschl, F., Berndt, A., Lubke, T., Ficner, R., *De novo sulfur SAD phasing of the lysosomal 66.3 kDa protein from mouse* 2009, Acta Crystallogr., Sect.D 65, 220

BL14.3

Manfred S. Weiss

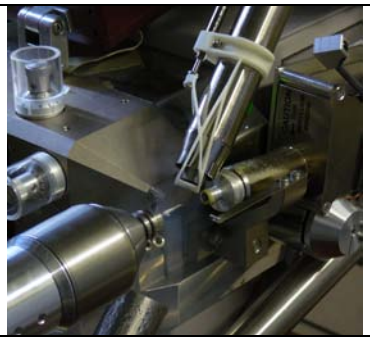


BL14.3 is a fixed energy beamline which is operated at an energy of 13.8 keV (0.89 Å). It can be used for *de novo* structure solution using anomalous phasing methods such as SAD utilizing heavy atoms like Pt, Hg, Au, Se and others. BL14.3 offers a unique experimental set-up to improve the diffraction properties of protein crystals. It is equipped with a HC1c dehydration setup, which can be used to improve the diffraction quality of crystals by controlled dehydration.

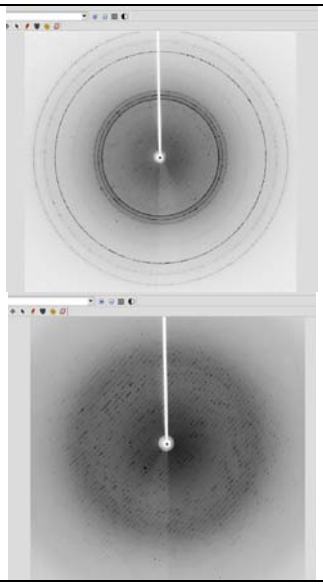
Energy [keV]	13.8
Wavelengths [Å]	0.89
Max. photon flux at sample [Phot/s/0.1A/0.05% BW]	4 x 10^{10} (13.8 keV)
Energy resolution [eV]	< 5
Goniometry	MARdtb
Sample automation	n/a
X-ray detector	Rayonics SX-165
Beam size [μm]	100 x 200
Achievable resolution [Å]	0.9
Maximum cell parameter [Å, at 2.0 Å resolution]	250
Average exposure time [sec]	3 - 30

Experimental possibilities:

- High performance *de novo* structure determination by SAD, SIRAS, MIRAS
- Crystal annealing with a remotely controlled cryo-shutter
- Controlled dehydration of protein crystals
- High resolution data collection



BL14.3 annealing device in operation

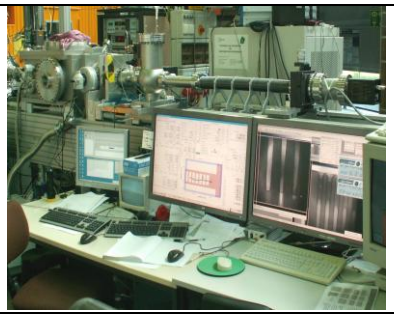


Macromolecular crystal diffraction image before and after annealing

Selected publication:
Kuettner, E.B., Kettner, K., Keim, A., Svergun, D.I., Volke, D., Singer, D., Hoffmann, R., Muller, E.C., Otto, A., Kriegel, T.M., Straeter, N., Crystal Structure of Hexokinase KlHxk1 of Kluyveromyces lactis: A MOLECULAR BASIS FOR UNDERSTANDING THE CONTROL OF YEAST HEXOKINASE FUNCTIONS VIA COVALENT MODIFICATION AND OLIGOMERIZATION (2010) J.Biol.Chem. **285, pp.** 41019-41033

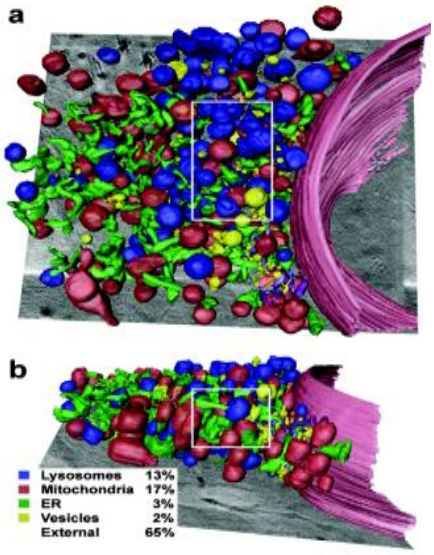
U41 X-ray Microscope

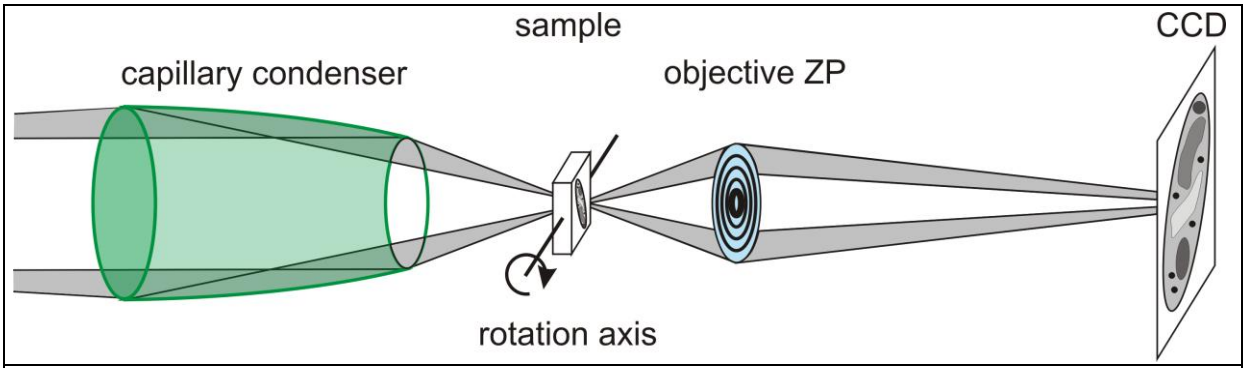
Gerd Schneider
Peter Guttmann
Katja Henzler
Stefan Rehbein
Stephan Werner



The X-ray microscopy group at the Helmholtz-Zentrum Berlin specializes in the development and application of advanced X-ray microscopy, X-ray tomography and X-ray optics for the 10-nm scale characterization of the nanostructure, chemical nature, and composition of materials with high energy resolution. This world-class facility is comprised of a unique combination of state-of-the-art X-ray microscopy instruments, in-house X-ray diffractive optical development and staff members with expertise in microscopy, physics, biophysics and chemistry to address national needs and technical challenges that impact materials, energy and life sciences.
The full-field cryo transmission X-ray microscope provides unique capabilities for high resolution X-ray imaging. It permits tomography of cryogenic samples on flat sample holders as well as spectromicroscopy studies with high energy resolution $\Delta E/E=10^{-4}$ at nanoscale lateral resolution.

Type	Cryo full-field transmission X-ray microscope
Photon energy range	0.25 – 1.5 keV
Energy resolution	10^{-4}
Sample temperature	- 170° C - room temperature
Sample tilt	±80°
X-ray sourc	U41 undulator
3D spatial resolution	25 nm
2D spatial resolution	10 nm
Monochromator	SGM





Selected publications:

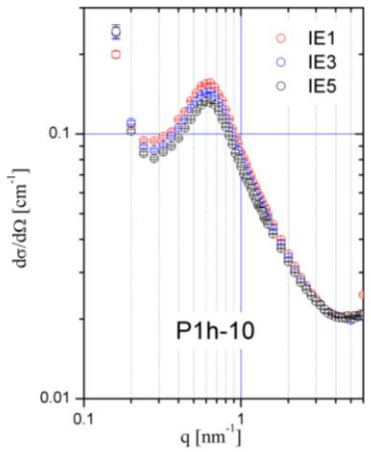
1. S. Rehbein, S. Heim, P. Guttmann, S. Werner, G. Schneider, *Ultrahigh-resolution soft-x-ray microscopy with zone plates in high orders of diffraction*, Phys. Rev. Lett. **103**, (2009) 110801
2. G. Schneider, P. Guttmann, S. Heim, S. Rehbein, F. Mueller, K. Nagashima, J.B. Heymann, W.G. Müller, J.G. McNally, *Three-dimensional cellular ultrastructure resolved by X-ray microscopyNature Methods* **7** (2010), 985-987
3. P. Guttmann, C. Bittencourt, S. Rehbein, P. Umek, X. Ke, G. Van Tendeloo, C. P. Ewels and G. Schneider, *Nanoscale spectroscopy with polarized X-rays by NEXAFS-TXM*, Nature Photonics **6** (2012), 25-29

ASAXS

Armin Hoell
(F-A2)
Günter
Goerigk

This small-angle X-ray beamline is run in close cooperation of F-A2 and F-I2. It provides access to measurements of anomalous SAXS (ASAXS; [1]). Within the realm of soft matter research, tremendous quantitative information about chemical concentrations in highly diluted chemical solutions can be obtained by ASAXS [2]. Moreover, ASAXS is highly suited to analyse polyelectrolytes in solution. Further scientific topics include organic/inorganic hybrid colloid particles for catalyst applications, bio-membranes serving as nano-reactors for the synthesis of semiconductor and magnetic colloids, and proton-conducting membranes form fuel cell technologies.

Type	Fixed exit double crystal monochro-mator, 2nd crystal sagittal focusing Si-111: $\Delta E/E = 2 \cdot 10^{-4}$
Photon energy range	3.8—15 keV, up to 26.7 keV without mirrors
Focusing mirror optics	Si and Rh tracks on mirrors
Beam size	~ 1 x 0.4 mm^2
Flux	$2 \cdot 10^{12}$ ph/sec/mm^2
Detectors	Ar/CO_2-MWPC 2d-detector (200 x 200 mm2 resolution 0.2 mm) CCD, diameter 165mm, resolution 80µ
q-range	8 keV: 0.09 - 6 [nm^{-1}]
Sample environment	Thermostat:: -20 – 95°C
Transmission accuracies	10^{-4}



ASAXS measurements on Rb counter ions distributed around PAA at 3 energies at the K-absorption edge of Rb at 15200 eV.

Developments:

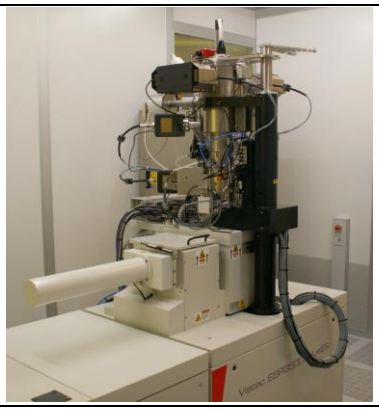
Improvement of *q*-resolution
Improvement of energy resolution
New detector concepts
Sample environment, shear cell, LS...

Selected publications:

1. Hoell, A., Zizak, I., Bieder, H., Mokrani, L., Patent DE 10 2006 029 449, 2006.
2. Goerigk G, Huber K, Schweins R. J. Chem. Phys. 2007; 127: 154908.
3. Haas, S., Hoell, A., Wurth, R., Rüssel, C., Boesecke, P., Vainio, U., Phys. Rev. B 2010; 81: 184207.

Electron Beam Writer

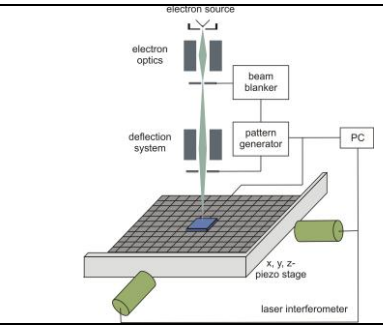
Stefan Rehbein
Stephan Werner
Gerd Schneider

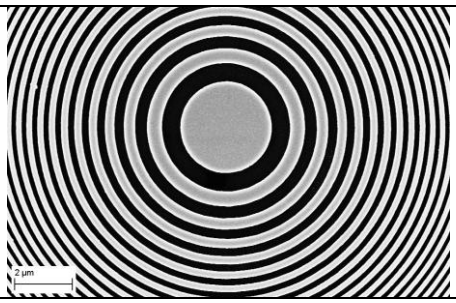


The X-ray microscopy group in the institute Soft Matter and Functional Materials operates a state-of-the-art electron beam writer from VISTEC (type EBPG 5000+ ES). With its small electron beam size in the range of few nanometers and the high electron energy of 100 keV, lithography with nanometer precision is possible. Arbitrary pattern can be computer generated, converted into machine readable format and finally exposed. The writing field size without moving the wafer stage is 256 µm at 100 keV electron energy. Larger areas can be exposed by stitching the writing fields using the laser interferometer controlled wafer stage. Under these conditions maximum areas of 4 inch can be exposed, depending on the writing time which increases with smaller step sizes. Another advantage of our e-beam tool is the high overlay precision in the 10 nm range which permits to stack processed layers. The application fields for the new e-beam system are diffractive X-ray optical elements, for example high-resolution Fresnel zone plates for X-ray microscopy or advanced monochromator gratings.

Specifications of the VISTEC EBPG 5000+ ES

Electron energy	50, 100 keV
Writing field size	256 µm (100 keV)
Beam current	100 pA – 100 nA
Pattern generator frequency	25 MHz
DAC	16 Bit (main-field), 14 Bit (sub-field)
Spot size	2.2 nm (100 keV)
On-axis resolution in resist	8 nm
Laser stage resolution	0.6 nm
Wafer size	up to 4 inch





Selected publications:

1. S. Rehbein, S. Heim, P. Guttmann, S. Werner, G. Schneider, *Ultrahigh-resolution soft-x-ray microscopy with zone plates in high orders of diffraction,* Phys. Rev. Lett. **103**, (2009) 110801
2. S. Werner, S. Rehbein, P. Guttmann, S. Heim, G. Schneider, *Towards high diffraction efficiency zone plates for X-ray microscopy Microelectron*, Microelectron. Eng. **87** (2010), 1557-1560
3. S. Rehbein, P. Guttmann, S. Werner, G. Schneider, *Characterization of the resolving power and contrast transfer function of a transmission X-ray microscope with partially coherent illumination,* Optics Express 20 (2012), 5830-5839

Soft Matter and Functional Materials

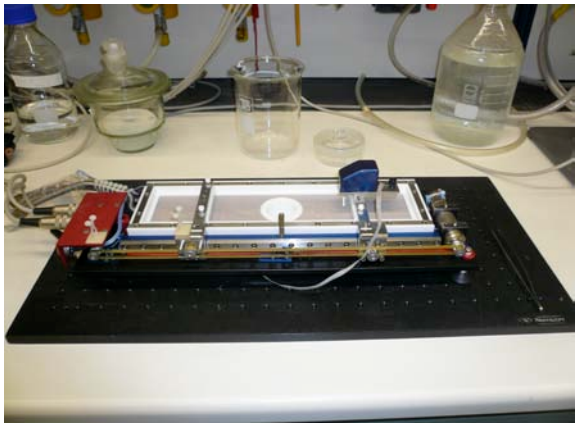
Laboratories

The BioLab: Thomas Hauß, Manfred Weiss

The BioLab is an essential service unit for the HZB user platform and provides on-site sample preparation and characterisation in parallel and complementary to neutron and X-ray scattering experiments The Wannsee branch of the BioLab is run by the Biophysics group and it is routinely used by neutron users and cooperation partners with the scientific background soft matter and biology, a community of 1/3rd of all neutron users. The BioLab offers biophysical, biochemical, and cell laboratories with a broad range of laboratory-based equipment. Specialised sample environments for neutron scattering experiments, especially complementary to neutron diffraction and reflectometry, SANS, SAXS, INS and QENS are developed. The expertise ranges from membrane biophysics and structural biology over protein dynamics to structure and function of interfaces. Significant achievements for the user support are sophisticated preparation methods for model membranes, a reliable and robust protocol for the preparation of “free floating bilayers” for neutron reflectivity, and a novel real-time (laser-neutron) pump-probe experiment to study modulation in protein dynamics by neutron scattering methods. Future developments will strengthen experiments under most physiological condition, establish in-situ sample characterisation like in-situ ATR-FTIR spectroscopy, and offer new characterisation methods like dynamic light-scattering and fluorescence microscopy.
The Adlershof branch of the BioLab is run by the MX-group and is currently being converted into a protein production facility for Structural Biology experiments. Once completed, it will be possible to perform all steps from cloning, heterologous bacterial expression, protein purification and characterisation as well as crystallization of proteins for X-ray diffraction experiments. The BioLab will support the research activities of the HZB MX-group but it will also remain available to other groups as well as external users.

Left: Fluorescence microscope with spot illumination

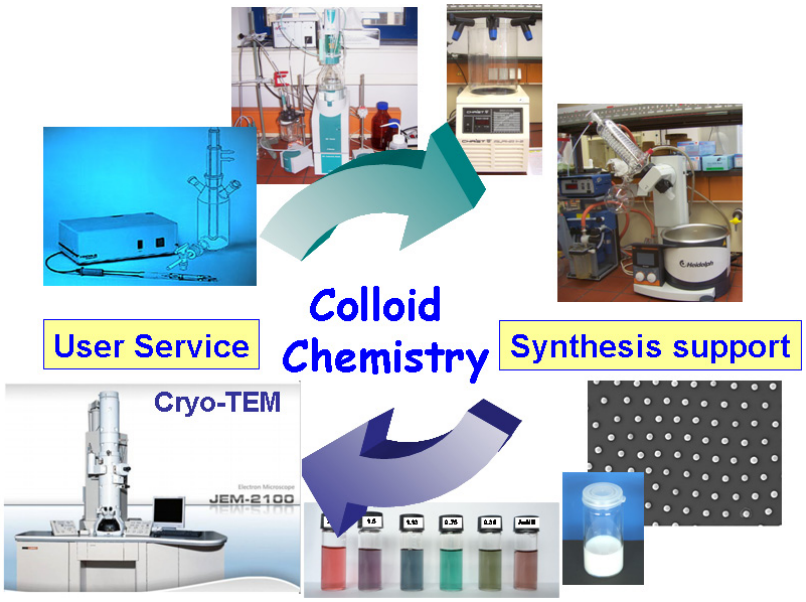
Right:
Cell Laboratory



Langmuir trough

Laser laboratory

The Chemistry Lab: Yan Lu



The chemistry lab has all the facilities for the synthesis and the characterization of colloidal suspensions, micelles, and supramolecular structures generated in aqueous suspension. The main task of chemistry lab is to give support for the users during their beam time in HZB. This includes not only supporting the user with full-equipped chemistry lab for sample preparation, but also providing cryo-TEM measurement service to the users. In addition, we supply users with professional guide for synthesis of different colloidal particles (from organic to inorganic particles).

All together we run the new cryo-TEM located at the Joint Laboratory of Structural Research (Adlershof), two synthesis labs (L130, L131), one sample purification lab (LS123), two sample preparation labs (V108 and UYH0343 (in Neutron Halle)), which can be accessed by our users. All the synthesis labs and sample preparation labs are equipped with fume hoods. Supplements, such as Millipore water, magnetic stirrer, thermostat, vortex mixer, balance and research pipettes, etc. are available in all labs. Storing samples either in refrigerator/freezer (~ 10°C) or ultra-low temperature freezer (~ -20°C) is possible during beamtime. At the Joint Laboratory of Structural Research, a new cryo-TEM is operated and the necessary service is provided to the users of this instrument.

Giving technique supports for synthesis is another core-task for the chemistry lab. Concerning the requests of the users, polymerization (such as emulsion polymerization, dispersion polymerization and Atom Transfer Radical Polymerization (ATRP)) can be conducted in the colloid chemistry lab. In addition, two UV-reactors are available in the synthesis lab that photo polymerization/reaction can be also carried out as requested. We supply the service of basic characterization of the colloidal samples as well (such as pH/conductivity measurement, auto-titration, zeta-potential etc.). Special sample treatment with high vacuum oven or freeze-drying is possible. In addition, this lab provides support by doing electron microscopic studies on colloids.

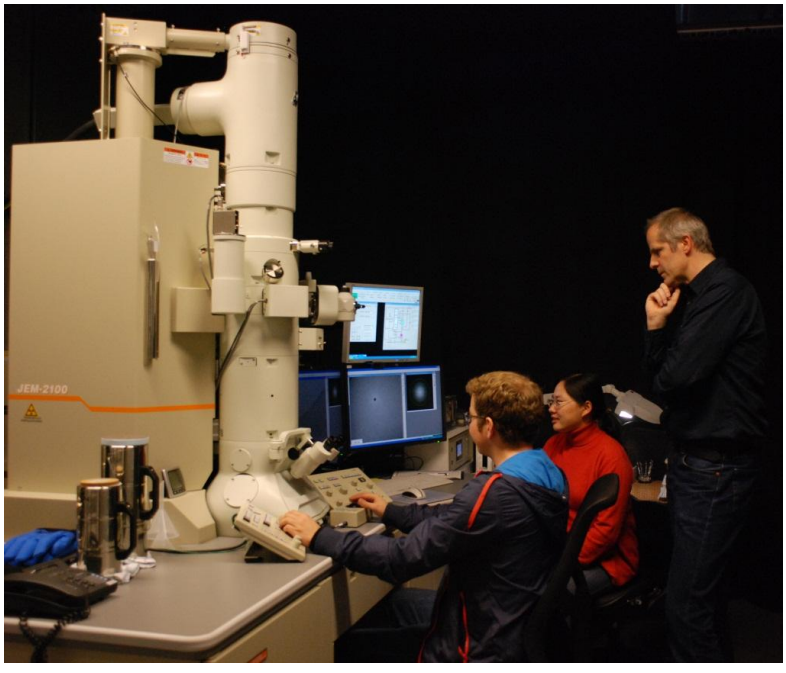
The combination of user service and professional technical support based on colloid chemistry provides an important research concept in the Institute of Soft Matter and Functional Materials.

Joint Laboratory for Structural Research

Soft matter systems consist of structures and structural units with sizes that span from the atomistic range to micrometers. Analysis of such systems hence requires a wide range of methods that have access to this range. Moreover, probes used for the structural analysis should be sensitive to the details of the system under consideration. The newly founded Joint Laboratory for Structural Research meets these demands and offers a wide range of methods summarized in the following table:

Partners	***Groups***	***Methods***
HU	Professorship for electron microscopy and structural research (W3)	HR-TEM of hybrid systems
	Prof. J.P. Rabe, PD S. Kirstein, Institut für Physik	AFM
	Prof. S. Kowarik, Institut für Physik	X-ray scattering and reflectivity
HZB	PD Dr. G. Schneider, Dr. K. Henzler, Soft Matter and Functional Materials	X-ray microscopy, E-beam lithography
	Prof. M. Ballauff, Dr. Y. Lu, Soft Matter and Functional Materials	Cryo-TEM
	Dr. G. Goerigk, Soft Matter and Functional Materials	SAXS, SANS
TU	Prof. Regine von Klitzing	Cryo-TEM

The laboratory for cryogenic transmission electron microscopy was established officially in December 2011 and is a part of the Joint Lab for Structural Research (JLSR) which is a collaborative project of the Helmholtz-Centrer Berlin (HZB), the Humboldt University of Berlin and the Technical University of Berlin. It is also used for the Z2 project within the Sonderforschungsbereich 951 on hybrid inorganic and organic structures (HIOS) which started in July 2011. The main concept of this laboratory is to provide structural information of soft matter and hybrid materials by transmission electron microscopy (TEM) and especially cryogenic TEM (cryoTEM). Combination of these investigations together with other methods available in the JLSR (e.g. X-ray and neutron scattering experiments @ HZB) provides information of materials on length scales from nm up to several µm. (Foto: © Raufeld/Gerd Metzner)

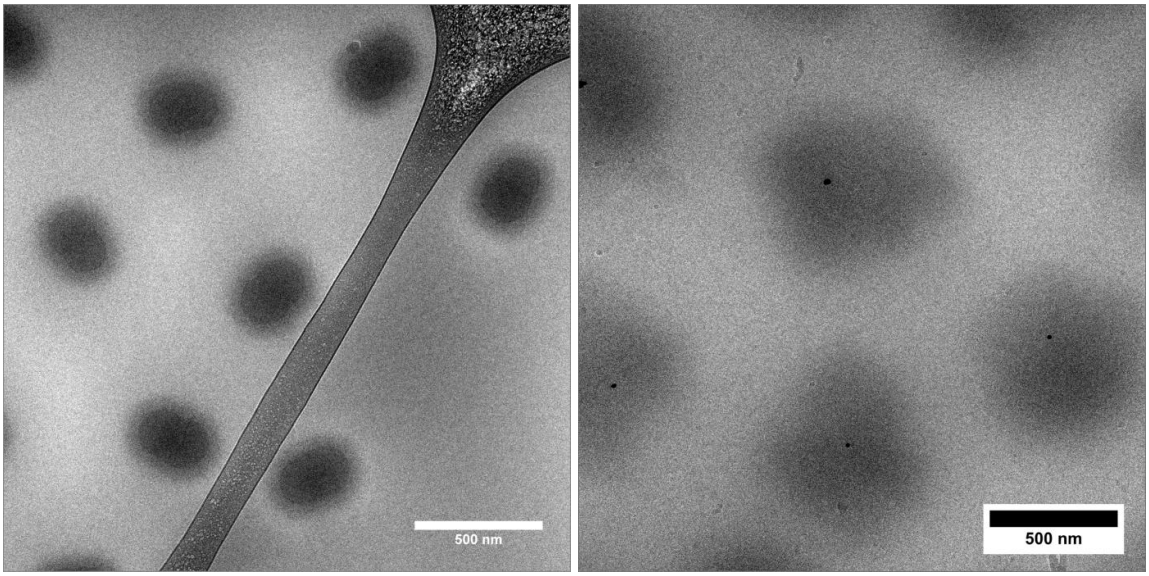


The laboratory for cryoTEM is equipped with a JEOL JEM 2100 microscope (JEOL Germany, Eching, Germany) with a LaB_6 thermoionic electron source and is optimized for investigations of vitrified samples. A TVIPS F416™ CMOS camera (TVIPS, Gauting, Germany) guarantees

high sensitivity for electron detection even at low dose conditions. The preparation lab includes a state of the art plunge freezing device for vitrifying liquid samples (FEI, Vitrobot Mark IV, FEI Deutschland GmbH, Frankfurt a. M., Germany). A high tilt cryo transfer holder enables investigations of the frozen samples at liquid nitrogen temperature within a tilt range from +80° to – 80° (Gatan GmbH, Munich, Germany). Automated tilt series acquisition software (TVIPS EMTOOLS™) make the cryoTEM a versatile tool for 3D investigation on colloidal particles including 3D reconstruction. A high-end multi-core computer with image processing and 3D reconstruction software is available.

Automated plunge freezing device (FEI Vitrobot™) for vitrifying particles in the liquid dispersed state. © Polzer

The field of research includes soft matter samples like synthetic polymer structures as well as hybrid organic inorganic materials. These materials are highly sensible to the electron beam and show severe structural changes under high vacuum conditions. Therefore vitrifying these particles in their dispersed state at liquid nitrogen temperature is an ideal approach to minimize both negative effects. Electron tomography of vitrified samples additionally provides 3-dimensional information about soft matter structures in their dispersed state. The data gained by this approach can be used for developing suitable models for fitting scattering curves of the respective structures.

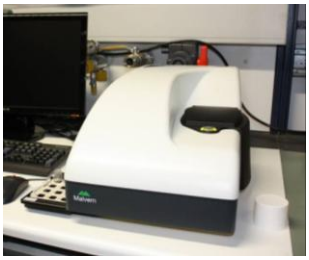


(left) CryoTEM micrograph of dumbbell shaped polystyrene core particles with a thermosensitive polymer network shell in vitrified water. (right) Poly(N-isopropylacrylamide microgels with a gold nanoparticle core. © Polzer

Colloid Lab: Daniel Clemens

The colloid lab is a user lab dedicated to the characterization with light and rheology. As a general rule, our equipment shall be available for guest groups, especially those scientists that have been granted beam time on the neutron or synchrotron instruments to allow for a complete and proper characterization of their samples. This opportunity is another manifestation of HZB's mission to offer "neutrons and more" to the users, which includes the support by the scientific and technical staff. Similarly to the regulations for the large scale research instruments, the access to the laser lab is subject to safety instructions prior to access. With these facilities we are near to a complete park of tools for the characterization of colloidal suspensions, micelles, and supramolecular structures generated in aqueous suspension.

The colloid lab hosts equipment for studies to measure physical parameters, complementary to the proposed experiments at the neutron and X-ray user instruments. It also serves HZB scientists in their daily "in-house" research. The lab is run by the colloid physics group within the Institute for Soft Matter and Functional Materials. The available laboratory methods comprise thermally controlled static and dynamic light scattering (SLS/DLS/DDLS). Both are situated in our large class IIIb laser laboratory (LS117) together with a commercial Malvern

light scattering instrument	**ALV 4000**	**ALV 125**	**Malvern Zeta-Sizer** Nano ZS 3600
picture			
type	DLS / DDLS, SLS	DLS	DLS , Zetapotential
light source	He-Ne-laser, 18 mW, 633 nm		frequency doubled DPSS laser, 50 mW, 532 nm
polarizer / analyzer	Glan-Thompson prism		
detector system	avalanche photodiode	photomultiplier	avalanche photodiode
size range	0.010 - 50 µm	0.050 - 50 µm	0.6 - 6000 nm
temperature range	10 - 80°C		2 - 90°C
Cuvettes / geometries	disposable glass cell, reusable quartz (1 - 2 ml sample volume)		quartz, plastic, dip cell for zetapotential measurements
		standard analytics with detector at 90°	standard: plastic, 10×10 mm²

Zeta-Sizer. Additionally we run three rheometers in a dedicated lab (LS210), one of which is foreseen to be adapted to the small-angle neutron scattering instrument V16. The group research on mesoscopic materials as colloidal suspensions, micelles and supramolecular structures is in this way fully cross-linked with the development of instruments as well as the accessibility to and service of supporting laboratory equipment.

Depending on the state of an accepted proposal, either short term or long term, we offer reduced (DLS, Zeta sizer) or full (+SLS, DDLS) access to our light scattering equipment. The corresponding table shall give an overview about the parameters of our light scattering spectrometers.

As another class of experiments that may be supported by lab experiments we offer for long term proposal users to benefit from our rheology instruments. This shall primarily complement the Rheo-SANS experiments on the VSANS instrument in order to couple structural data to rheological data.

Rheometers	**MCR 301**	**PAV** piezoelectric axial vibrator	**TR** torsional resonator
picture			
rheometer type & supplier	rotational rheometer (Anton Paar)	squeeze flow rheometer (IdM Ulm)	torsional resonator (IdM Ulm)
shear rate range	10^{-5} -10^{4} s^{-1}	-	-
frequency range	10^{-4} – 10^{1} Hz	10 – 3000Hz	13, 25 and 77 kHz
temperature range	-40 – 200°C ± 0.01°C	5 – 60°C ± 0.02°C	5 – 60°C ± 0.02°C
geometries	<u>cone-plate:</u> R = 25 mm; α=0.991° sample volume: 0.6 ml	<u>rings:</u> gap height: 10..200 µm sample volume: 100 µl	<u>cylinder:</u> f = 25kHz and 77kHz sample volume: 7 ml
	<u>coaxial cylinder:</u> gap width: 1.128 mm sample volume: 19 ml		<u>dumbbell:</u> f = 13kHz sample volume: 11 ml
	<u>coaxial double gap:</u> gap width: 0.42 mm and 0.46 mm; η < 0.2 Pa sample volume: 3.8 ml		

The Joint Berlin MX Laboratory: Uwe Müller, Manfred S. Weiss

Figure 1: Inauguration of the Joint Berlin MX-Laboratory

Over the past years, Berlin has become the German center of X-ray crystallography based structural biology, with currently 12 independently operating macromolecular crystallography groups.
Within this group of researchers, the HZB-MX beamlines are a natural condensation point for most of the experimental work carried out within the 12 groups. Even more, the proximity of the MX-beamlines to the laboratories of the groups may even be the major reason for the success of the groups. Consequently, in 2008 a more formal platform for collaboration has been established by the following institutes:

- Helmholtz Zentrum Berlin für Materialien und Energie
- Freie Universität Berlin
- Humboldt Universität zu Berlin
- Max-Delbrück-Zentrum für Molekulare Medizin in Berlin-Buch
- Leibniz-Institut für Molekulare Pharmakologie

These five institutes have founded the “Joint Berlin MX-Laboratory” as a new model for a multi-institutional research collaboration in Berlin.

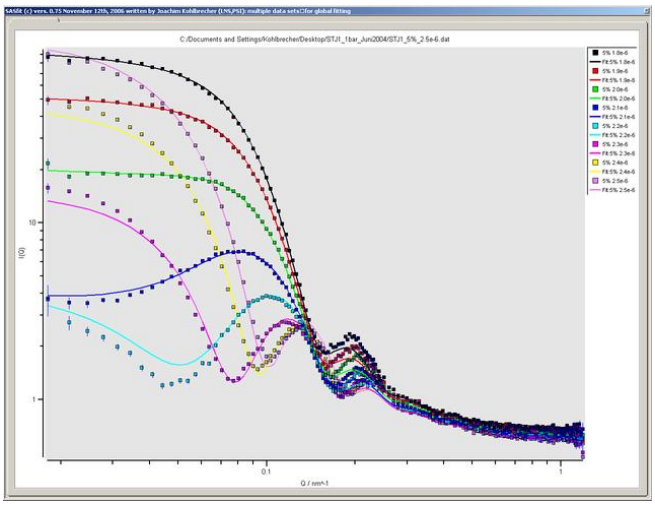
Within the next four years the participarting will work on a number of already initiated research projects, which were motivated by the collaboration partners and thus directly link the distinct research fields with each other. If possible the projects will be connected with each other, using the new possibilities available within this joint activity.
In addition to the research, the partners share the responsibility for the operation and future development of BL14.2-3 as well. First fund raising results could be achieved already through the submission of three joint research proposals, which did succeed in one BMBF-funded project, for the duration of three years. Within this project, the Freie Universität Berlin, HZB and the Russian Academy of science will work closely together within the field of structural biology of ribonucleoprotein complexes.


Figure 2: Meeting of 73 Berlin structural biologists at BESSY II during the 1. Joint MX-day in August 2010

The Theory Support Lab: J. Dzubiella,

The in-house theory group of the Soft Matter Institute F-I2 offers user-support for the analysis, fitting, and possible interpretation of (neutron and X-ray) scattering data. The latter may represent structure from a large variety of soft systems on different length scales, such as colloidal, polymeric, or molecular fluids. Possible collaborations between theory lab and users on the analysis of non-standard problems by advanced statistical methods and computer simulations are encouraged in the framework of long-term proposals.

At the instrument V16/VSANS data is recorded as "event-data" i.e., space and time coordinates [time-of-flight (TOF)] are detected and stored. With the software *egraph* the raw data is exported and read by the program *Mantid* for further TOF-data reduction (*http://www.mantidproject.org*) to provide the final scattering intensity *I*(*Q*). For the analysis of the reduced data the program *SASfit* is employed (*http://kur.web.psi.ch/sans1/SANSSoft/sasfit.html*). It offers a broad variety of form factors *P*(*Q*), ranging from those of spheres and cylinders, to more complex structures like solvated core-shell particles. The P(Q) can be freely combined for the convolution with a set of structure factors *S*(*Q*) to fit measured intensities, see Fig. 1. The *S(Q)*s offered by SASfit include common functions based on hard sphere or sticky-hard sphere potentials as well as more special ones like, e.g., of Hayter/Penfold type. If necessary for nonstandard fitting, own structure models can be programmed and added as a new plugin. Current efforts of the theory lab include extensions to offer structure factors for polydisperse, Yukawa-type, and multi-component mixtures. The theory lab offers consultation for the implementation of more specialized structures or numerical operations, such as nonlinear regression of intensity data.



Screenshot SASFit. A program for fitting elementary structural models to SAS data by J. Kohlbrecher and I. Bressler (Paul Scherrer Institut; http://kur.web.psi.ch/sans1/SANSSoft/sasfit.html)

Special software for data analysis of ASAXS experiments will be developed based on upgrading the data reduction software of the former dedicated ASAXS instrument JUSIFA, supplemented by special separation algorithms providing confidence tests for the inversion routines used for separation of the basic scattering functions.

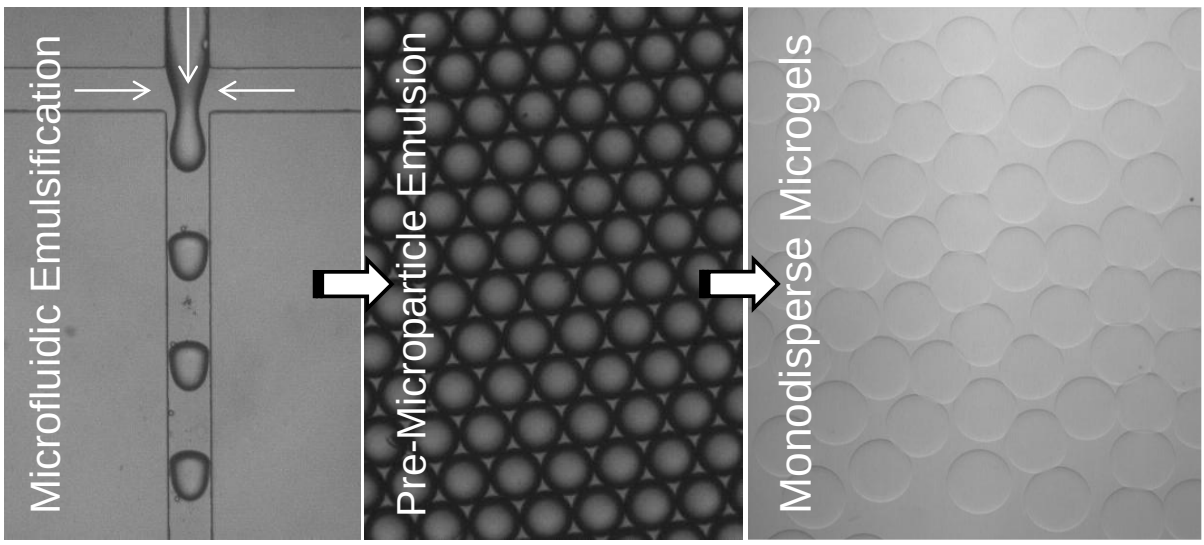
For more complex systems the theory invites users for collaboration within well-defined, long-term proposals. The theory lab has expertise in the application of typiycal statistical mechanics tools, like integral equation theory (IET) and density functional theory (DFT), as well as Monte-Carlo (MC), Brownian dynamics (BD), or molecular dynamics (MD) computer simulations for the description of fluid many-body structures.

Laboratory for Microfluidics: Sebastian Seiffert

Microfluidic devices are networks of micron scale channels integrated to perform functions. These functions can be divided into two broad classes. In one class, microfluidic devices perform chemical and biological assays by introducing cells, beads, and other reagents into the device and then merge, mix, and split them. In the second class, microfluidic devices fabricate fluid droplets of precisely controlled geometry, which then serve to synthesize microparticles. For this purpose, solutions or melts of monomers or crosslinkable polymers are introduced into the device, along with an immiscible carrier phase; the devices disperse these solutions into equally sized micro-droplets, which can then be solidified by polymerization, crosslinking, or crystallization, as illustrated in the figure.

The principle of the microfluidic drop formation can be explained using a water faucet as an example: if a faucet operates a low flow rate, water drips out one drop at a time. The drop size is determined by a balance between the surface forces of the hanging drop and its weight, and therefore depends on the surface tension of the fluid and the size of the faucet. Since both these parameters are constant, all drops exhibit a narrow size distribution. The same principle is employed in microfluidic channels, such that the droplet size obtained in these channels is highly controllable. Microfluidic devices also offer versatile means to form complex structures such as non-spherical droplets, anisotropic droplets, or multiple-emulsion “droplets-in-droplets”. These structures can be retained by subsequent droplet solidification, typically achieved through rapid on-chip polymerization, thereby yielding monodisperse particles with complex architecture.

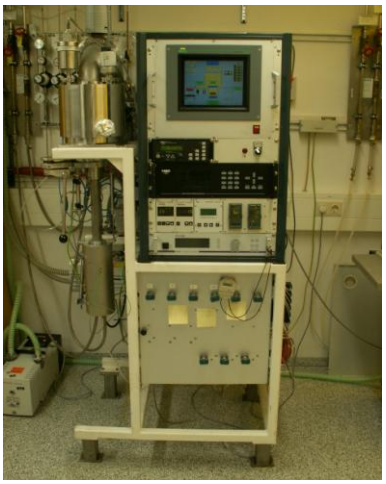
Another application of microfluidic channels encompasses their implementation in scattering experiments. The strategy for this is to place an array of microchannels into an x-ray or neutron beam and to probe the species of interest while flowing through the channels. The channels to be used for this purpose can either have constant dimensions, or they may exhibit varying width and height, allowing the samples to be probed in uni- or biaxially deformed states. As an alternative, a semi-static method aims to use a microfluidic platform which resembles a “parking lot for droplets”, allowing single deformable samples such as droplets or microgel particles to be fixed in addressable positions. Subsequent longterm-observation can serve to monitor ageing or relaxation processes.



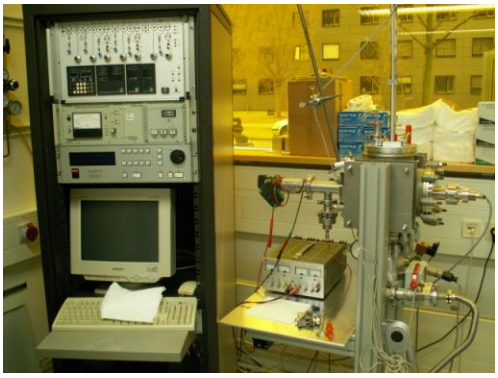
Microfluidic fabrication of polymer microgel particles. A flow focusing microfluidic device serves to form monodisperse pre-micrpoparticle droplets, which then serve to template monodisperse microgels.

The Nano Lab: Stephan Werner, Stefan Rehbein, Gerd Schneider

The Nano laboratory for X-ray microscopy optical elements is operated by the X-ray microscopy group at the WCRC in Adlershof. It is equipped with advanced tools specialized for nanofabrication of high resolution zone plates, phase rings and test structures with nanometer size. Tools for the preparation of thin film polymer layers such as a high speed spin-coater as well as vacuum and non-vacuum ovens for pre- and post-backing of the polymer layers are available. An electron cross-linking unit is used for the post hardening of polymer layers to increase their stability in subsequent chemical-mechanical polishing processes. Two extractor hoods contain all the required equipment for room and cryo temperature development of thin film e-beam resists, electro-plating of nickel and gold nanostructures and KOH wet etching processes of silicon substrates. An advanced reactive ion etcher (RIE) for low-pressure plasma etching transfers the pattern generated by the e-beam writer into metal masks and polymer layers. Additionally, a profilometer for accurate height measurements of thin layers (also on the fabricated structures) is installed as well as a light microscope working in transmission or reflection geometry which is used for quality inspections of the X-ray optical elements.



Reactive ion etcher for plasma etching of metal masks and polymer layers.



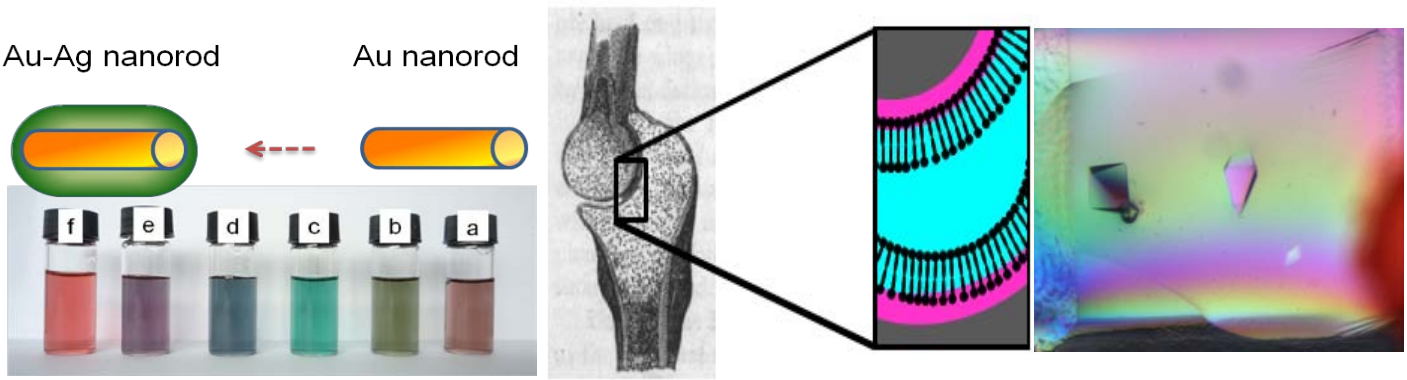
Electron cross-linking tool for post hardening of polymer plating molds.

Tools for quality inspection and thickness measurements of manufactured zone plates: profilometer (left) and light microscope (right).

Extractor hood equipped with high speed spin coater (right) and wet etching tools (left).

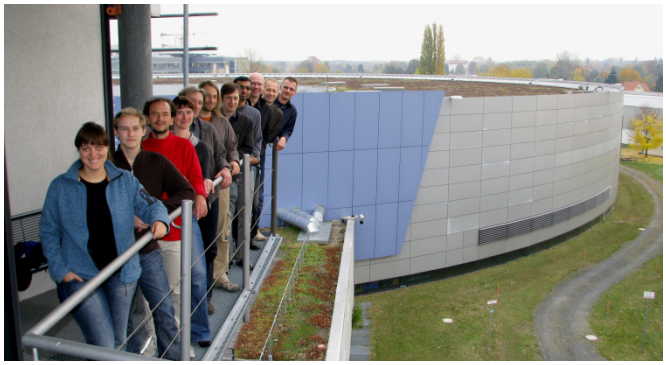
Soft Matter and Functional Materials

Research

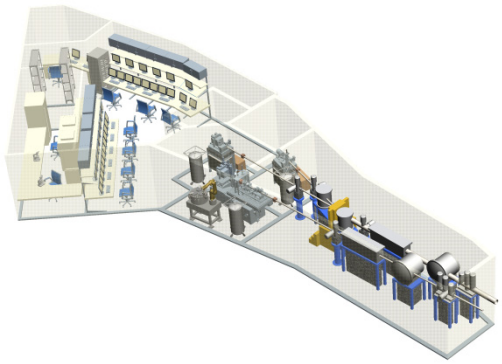


Macromolecular Crystallography (MX)

Uwe Müller, Manfred S. Weiss



Within the past two decades, structural biology has created enormous impact on all biological, biochemical and biomedical research fields. In particular Macromolecular Crystallography (MX) has matured from a few dozen scattered research groups to a field which is now present at every major university and research institution around the world. This is one of the reasons, why MX beamlines can now be found nowadays at all modern third generation synchrotron sources. The BESSY II based MX research group, which is led by Uwe Mueller and Manfred S. Weiss, operates and develops three MX-beamlines and consists currently of eight researchers and three technical staff members (picture).



The MX activity has been started at BESSY II in strong collaboration with the Free University Berlin in the year 2000. Since 2003 all experimental stations have been used within the regular user operation scheme of BESSY II and have produced more than 500 new protein structures, which are deposited at the Protein structure database PDB (www.rcsb.org/pdb).

The major task of the MX-group is the provision and the technical as well as scientific support of the experimental resources at a constant high-quality level for the international user community.

The HZB-MX user community currently consists of 43 international research groups, which are running 16% of all photon based research projects at BESSY II. Among these are 12 Berlin based MX-groups. In 2009 the “Joint Berlin MX Laboratory”, a research collaboration between FU-Berlin, the HU-Berlin, the Max Delbrück Centrum Berlin, the Leibniz Institute for Molecular Pharmacology (FMP) and the HZB has been founded to organize the usage, maintenance and development of the MX-beamlines on a collaborative basis.

The BESSY-MX group is also engaged in several research collaborations with regard to the “Joint Berlin MX-Laboratory”, as well as in a number of in-house research project in the field of MX. The two main pillars of these in-house research activities are MX methods development and structure-function relationship of enzymes which are capable of the degradation of environmentally critical soil pollutants.

Group publications (selection):

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- Klein N., Senkovska, I., Gedrich K., Stoeck U., Henschel, A., Mueller U., Kaskel S. (2009) Eine mesoporöse Metall-organische Gerüstverbindung. Angewandte Chemie 121, 52, 10139-10142.
- Merli, A., K. Manikandan, É. Gráczer, L. Schuldt, R. K. Singh, P. Závodszky, M. Vas & M. S. Weiss. Crystallization and Preliminary X-ray Diffraction Analysis of Various Enzyme-Substrate Complexes of Isopropylmalate Dehydrogenase from *Thermus thermophilus*. (2010). Acta Cryst. F66, 738-743.
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- Paithankar, K. S. & E. F. Garman (2010). Know your dose: RADDOSE Acta Cryst.. D66, 381-388.
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- Karuppasamy, M., A. Geerlof, A. Zozulya, D. I. Svergun & M. S. Weiss. Structural Studies on the Enzyme Complex Isopropylmalate Isomerase (LeuCD) from *Mycobacterium tuberculosis.* (2011). Proteins 79, 35-49.
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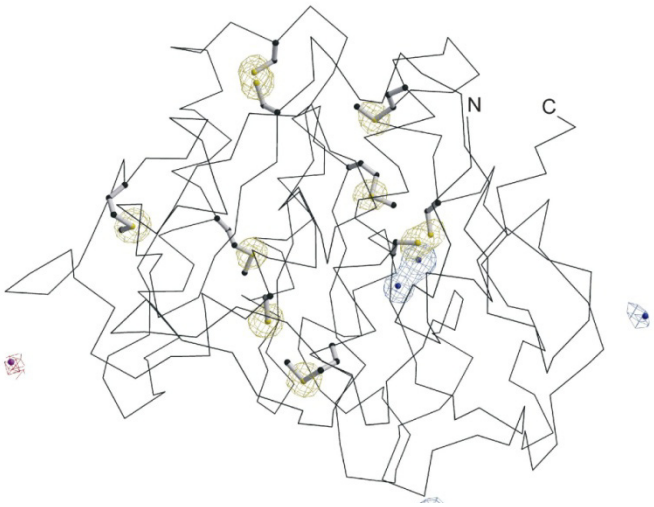
- Unge, J., C. Mueller-Dieckmann, S. Panjikar, P. A. Tucker, V. S. Lamzin & M. S. Weiss (2011). On the Routine Use of Soft X-Rays in Macromolecular Crystallography, Part V – Molecular Replacement and Anomalous Scattering. Acta Cryst. D67, 729-738.

Listing of all coworkers

- Dr. Martin Bommer
- Ronald Förster
- Michael Hellmig
- Michael Krug
- Dr. Uwe Mueller
- Dr. Karthik Paithankar
- Dr. Sandra Pühringer
- Michael Steffien
- Dr. Monika Ühlein
- Dr. Manfred S. Weiss

Phase determination Using the Anomalous Signal from Sulfur Atoms

Manfred S. Weiss



C_α-trace of the enzyme proteinase K. The superimposed anomalous difference electron density clearly shows the positions of all S-atoms (in yellow) of the protein, as well as the bound cations (in blue) and anions (in red).

In macromolecular structure determination, the solution of the crystallographic phase problem remains an unsolved issue in the whole process. Over the years, many methods have been developed to derive phases from the diffraction intensities, all of which are grounded upon a modification of the macromolecules under investigation. Since nearly every protein contains sulphur-atoms and every nucleic acid contains phosphorous atoms, it may be anticipated that the anomalous scattering of the S- and P-atoms intrinsically present in the macromolecules, can be used for phase determination. If such an approach would turn out to be commonly applicable, the phase problem in macromolecular crystallography would cease to exist. In the MX-group at the HZB, we are developing tools overcome the major difficulties associated with this **S**ulphur **S**ingle Wavelength **A**nomalous **D**iffraction (S-SAD) approach.

The main difficulty associated with S-SAD is that the achievable signal is extremely small [1]. The S-SAD related research in the MX-group at the HZB is focussed on two pillars: first, to increase the signal by collecting diffraction data at slightly longer than usual wavelengths (1.8-2.2 Å) and second to measure the intensity differences with a very high precision (about 1%).

A data collection experiment at longer wavelengths is in principle not any different than an experiment at short wavelengths. However, due to the fact that absorption becomes a more and more severe problem as one goes to longer wavelengths, such an experiment has to be carefully planned and carried out. At present, it seems that a data collection wavelength of about 2.0 Å yields the highest anomalous signal-to-noise ratio [2]. With better crystal mounting procedures and better data reduction tools available, however, the hope is that this may shift to even longer wavelengths, where the signal is further increased.

In order to collect diffraction data to the highest possible precision, a number of tools are available at the HZB-MX-beam lines. For instance, BL14.1 is equipped with a kappa-goniometer, which enables the experimenter to orient the crystals and various data collection strategy options help to devise the optimal data collection strategy.

To date, only about 50-100 macromolecular structures have been determined using the S-SAD approach. Two examples are given in references 3 and 4. The current status is that macromolecules crystallized in high-symmetry crystals with a relatively small asymmetric unit and good diffraction properties are amenable to structure determination by S-SAD. We hope, though, that the method can be further developed in order to push the boundaries towards lower symmetries and larger structures.

[1] K. Djinović Carugo *et al.* (2005). *J. Synchr. Rad.* **12**, 410-9.
[2] C. Mueller-Dieckmann *et al.* (2005). *Acta Cryst.* **D61**, 1263-72.
[3] M. S. Weiss *et al*. (2004). *Acta Cryst.* **D60**, 686-95.
[4] K. Lakomek *et al.* (2009). *Acta Cryst.* **D65**, 220-8

Phase Determination Using the UV-Light Induced Radiation Damage

Uwe Mueller

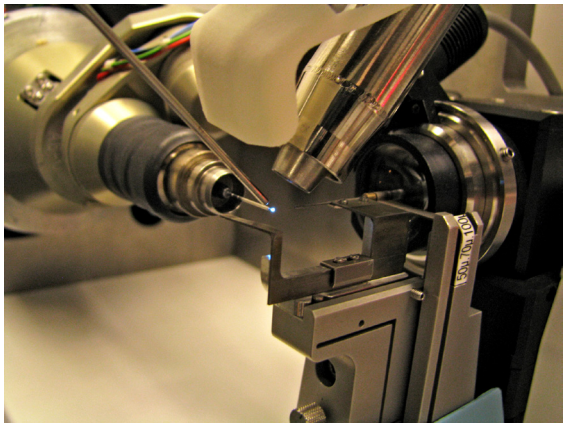


Figure 1: UV-laser setup at BL14.1 during irradiation of an thaumatin crystal

After the collection of an X-ray diffraction data-set from a macromolecule crystal the solution of the so-called “crystallographic phase problem” is the major task, which must be resolved. In order to achieve this, a growing number of methods exist, which we are aiming to extend with the further development of the UV-based radiation induced phasing (UVRIP) method [1]. This experimental technique is focused on specific structural changes of cystine-containing protein crystals, which is due to the irradiation of the specimen with highly-intense UV-radiation (Figure 1). The structural changes can be used to work out a single isomorphous replacement (SIR)-like phasing scheme, which can lead to precise experimental phase information and thus to the access to the three dimensional structure (Figure 2). For this, a native data-set has to be collected before UV-exposure and compared with a second data set collected after the UV-irradiation of the same crystal. At the HZB-MX beamline BL14.1, we have installed all required instruments to carry out such experiments [2,3] and are providing this to the user community. Within this research project, we aim to develop this method to reduce the existing requirements in terms of minimal data-set resolution and to investigate alternative specific damage sites within a macromolecular crystal.

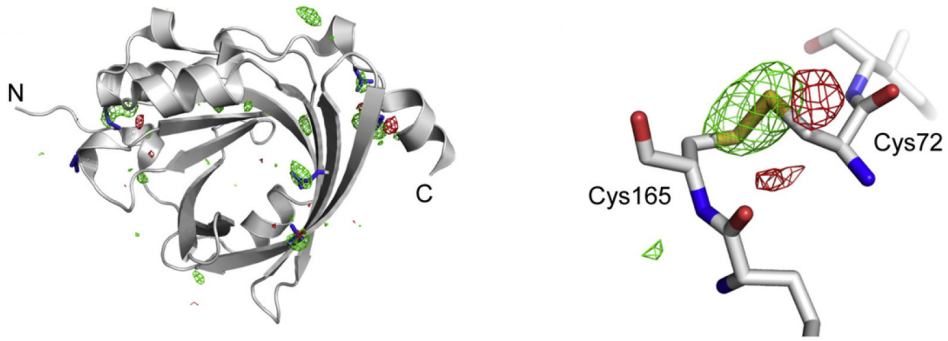


Figure 2: Specifically damaged disulfide-bridges of the protein α_1-acid glycoprotein

[1] M. H. Nanao *et al*. (2006). *Structure* **14**, 791-800.
[2] A. Faust *et al.* (2010). *J. Appl. Cryst.* **43**, 1230-1237.
[3] D. L. Schoenfeld *et al.* (2008). *J. Mol. Biol.* **384**, 393-405.

Diffraction-Based Screening to Rapidly Characterize Biological Crystals in their Native Environment

Karthik S. Paithankar

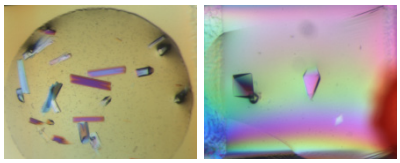


Figure. 1 Crystals of the proteins insulin and lysozyme

One of the challenges in macromolecular crystallography is the identification of suitable crystals for diffraction data collection. In the past decade structural genomics projects and pharmaceutical companies have successfully created pipelines to produce and purify proteins in a rapid manner. Crystals from these proteins are grown in crystallization trays being able to harbour 96, 192, or 288 different experimental conditions, using automated liquid handling systems (Figure 1). So far crystals are pre-selected by optical imaging but this method does not provide any information about diffraction properties of the specimen. Using a microscope it is often not possible to distinguish salt and other small molecule from protein crystals. In addition, the first crystals obtained in these experiments are usually very small and fragile.

Figure 2 A crystallization plate with samples ready for X-ray analysis

One alternative to the classical imaging method is the rapid identification of crystallization conditions that provide diffraction quality crystals *in situ* at room temperature by X-ray diffraction [1]. Within our group, we are developing and applying such methods by exposing the crystallization trays directly to X-ray beam at room temperature. using a robot.

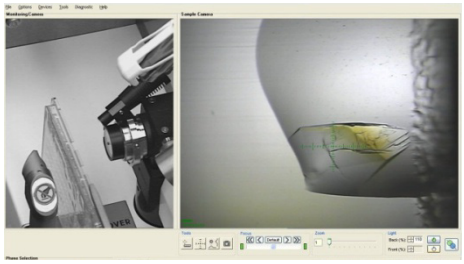


Figure 3 A view of the crystal after X-ray irradiation

The hardware implementation consists of a 6-axis industrial robot arm, which can handle crystallization plates (Figure 1). The crystal, which is grown inside the drop in the plate, can thus be directly exposed by the X-ray beam (Figure 3). Only few X-ray images are required to gain information about the crystal characteristics, such as the unit cell, symmetry and mosaicity.

Thus, instead of undergoing the long and exhaustive process of growing hundreds of crystals and testing each crystal in the X-ray beam, a single crystallization tray can be exposed to find the diffraction characteristics of all freshly grown crystals at ones.

Note in addition: Recently, a joint BMBF-funded research project has been initialized between the HZB and the Russian Academy of Sciences and the Freie Universität Berlin to develop this method.

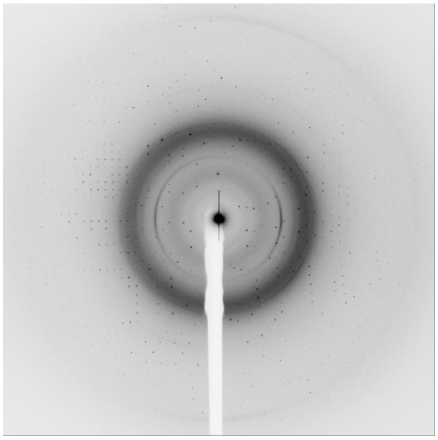
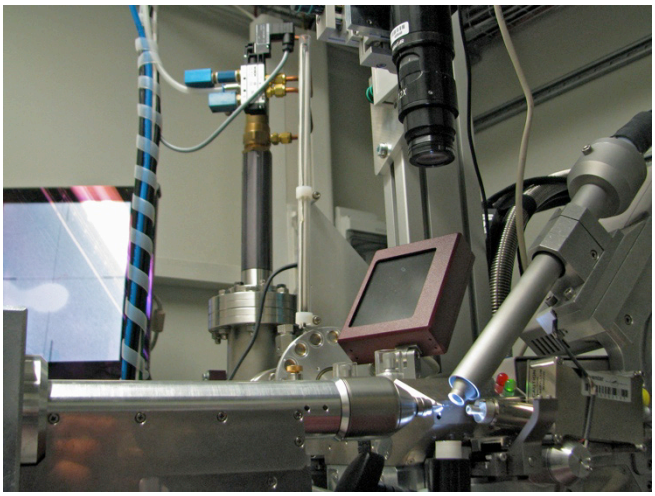


Figure 4 Diffraction image from a protein crystal sitting in a plate

[1] Jacquamet *et al.* (2009). *J. Synchr. Rad.* **16**, 14-21.

Controlled crystal dehydration using the HC1c device installed at BL14.3

Manfred S. Weiss, Uwe Mueller



Endstation of the BL14.3 with HC1c-dehydration system setup

Many biological macromolecules yield crystals, which display significant inherent disorder, and as a consequence diffract X-rays rather poorly. One approach to increase the crystalline order is to dehydrate the crystal in a controlled manner before shock-cooling it to 100 K for storage or for data collection. One of the first commercial devices for this was the Free Mounting System FMS developed by Kiefersauer and colleagues [1]. However, due to its rather difficult and cumbersome operation and due to its prize, the FMS never became very popular in the MX community. Recently, a new device (HC1b) has been developed by Sanchez-Weatherby and colleagues at the EMBL Grenoble, which allows for a much easier utilization of this technique [2,3]. Since January 2011, a HC1c device is installed on BL14.3 (Figure 1). It is very easy to operate and allows the user to improve the diffraction limit of a crystal by controlled dehydration, to determine optimal cryo-cooling conditions, to determine optimal soaking conditions for macromolecular crystal and to collect diffraction data at ambient temperature.

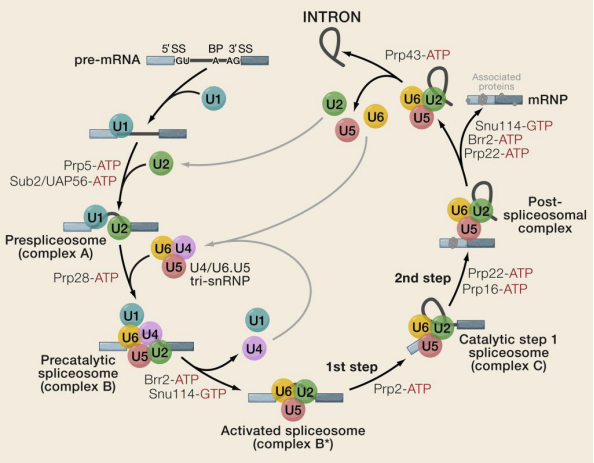
As part of our in-house research program we are aiming at developing protocols for the dehydration of macromolecular crystals in order to improve the internal order of the crystal. Concomitantly, the X-ray diffraction power of the crystals is improved. Such protocols include stepwise reduction of the ambient humidity, but the can also include re-hydration steps. Another aspect of utilizing the HC1c is the development of efficient procedures for mounting macromolecular crystals without surrounding mother liquor: dry-mounting. Dry-mounting is of particular importance when data collection at longer X-ray wavelengths is attempted for instance for sulphur-SAD phase determination. Absorption of X-rays is a big problem for such experiments; hence the crystals should be mounted with as little mother liquor or cryo-solvent as possible. The idea of dry-mounting was developed by Kitago and colleagues [4,5], but the semi-automated device installed at the Photon Factory (Tsukuba, Japan) is difficult to use and requires a lot of experience and practice. Recently, we were able to demonstrate that using the HC1c, dry-mounting is possible as well, but it still requires the application of cryo-solvent around the crystal in order to inhibit the formation of ice. Procedures are being developed to efficiently protect the crystal while at the same time shock-cooling it to 100 K in liquid nitrogen.

[1] Kiefersauer *et al*. (2000). *J. Appl. Cryst.* **33**, 1223-1230.
[2] Sanchez-Weatherby *et al.* (2009). *Acta Cryst.* **D65**, 1237-1246.
[3] Russi *et al*. (2011). *J. Struct. Biol.* **175**, 234-243.
[4] Kitago et al. (2005). *Acta Cryst.* **D61**, 1013-1021.
[5] Kitago et al. (2010). *J. Appl. Cryst.* **43**, 341-346.

Structure and Function of Protein Complexes from the Spliceosome

Sandra Pühringer

The spliceosome is a large and highly dynamic RNA-protein machine that deletes unimportant (so called “non-coding”) regions from the DNA – a process called splicing. For each splicing event, a spliceosome is assembled *de novo* on the pre-mRNA, extensively remodeled and, after the deletion of the non-coding regions, disassembled in an ordered fashion (Figure).



Schematic representation of the spliceosomal assembly. Adapted from [1].

As in all complex cellular processes splicing is also known to be a potential source of errors during gene expression. Several diseases are known that are directly linked to errors during pre-mRNA splicing (e.g. retinitis pigmentosa) and even specific forms of cancer are potentially caused by spliceosomal anomalies.

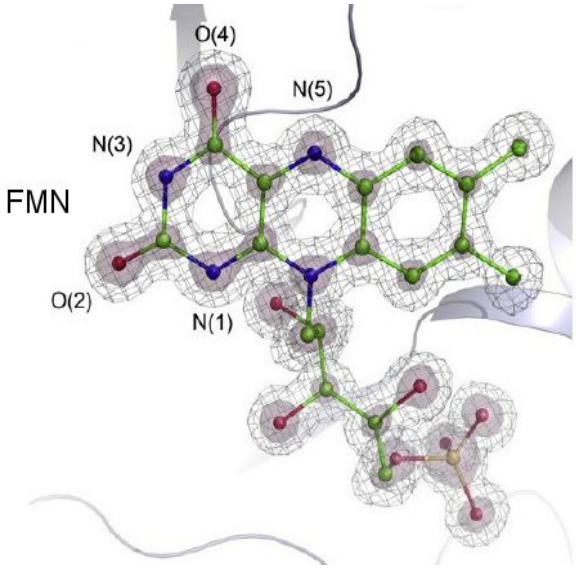
In humans, about 200 proteins, five small nuclear RNAs and the pre-mRNA intimately participate in the splicing process. During spliceosome assembly, catalysis and disassembly, many protein-protein, protein-RNA and RNA-RNA interactions are formed and broken in a controlled manner [1]. Presently, the exact sequence of remodeling steps and the functional roles of individual components of the spliceosome during assembly and catalysis are not fully understood.

In close collaboration with member institutes of the Joint Berlin MX-Laboratory, we screen for low molecular weight substances, which hyper-stabilize crucial protein-protein interactions that normally form only transiently during splicing. In a radically new approach we are aiming towards the identification of compounds, which bind at the interface of protein complexes and have the potential to hyperstabilize these interfaces. Such compounds would help in biochemical studies of spliceosomal assemblies since they arrest the molecular machine at a certain stage and they would also provide the basis for new therapeutic approaches. It must be anticipated that for such a project a very large number of diffraction data sets must be collected in order to identify suitable compounds. Therefore, the environment created by and the possibilities within the Joint Berlin MX-laboratory are ideally suited to carry out this project.

[1] M. C. Wahl *et al*. (2009). *Cell*. 1**36**,:701-718.

Xenobiotic Reductase A of Pseudomonas Putida

Michael Krug



The FMN molecule in the active site of XenA and its corresponding electron density. Oxygen atoms are shown in red, nitrogen atoms in blue, carbon atoms in green and phosphorus in yellow

Xenobiotics are substances that can be found in biological systems but are not supposed to be there (xenos=foreign, bios=life). Due to continuous evolutionary pressure, microbes have developed a diversity of pathways to cope with and to degrade xenobiotics such as aromatic and hetero-aromatic compounds.

Pseudomonas putida 86 was isolated near a coal tar factory (Rütgerswerke, Castrop-Rauxel) in Germany. Xenobiotic reductase A (XenA) of *P. putida 86* is involved in the degradation of quinoline, an ubiquitous soluble, heteroaromatic pollutant with cancerogenic properties. XenA contains flavin mononucleotide (FMN) resulting in a yellowish color of the enzyme. FMN is produced from vitamin B_2 in the cell and plays an important role in reactions comprising electron transfers.

XenA catalyzes the reduction of various substrates. The reaction of XenA can be divided into two half-reactions. In the first half-reaction, the oxidized enzyme is reduced by NADH, a reducing agent of living cells. In the second half-reaction, the enzyme itself can reduce different substrates whereby it gets oxidized. Atomic resolution crystal structures of XenA showed that the redox-sensitive bond lengths of the FMN are neither typical for oxidized nor for reduced flavins but are in between the distances expected for either one of the oxidation states. It is therefore likely that the synchrotron radiation reduced at least parts of the protein molecules during data collection leading to a mixture of oxidized and reduced molecules in the crystal.

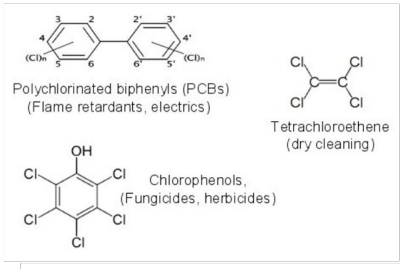
In order to get a better understanding of how the enzyme works, it is essential to obtain insights into the structural differences between the oxidized and the reduced state of XenA. Using adapted data collection and data merging strategies, we are attempting to separate the data representing the oxidized state of the enzyme from those representing the reduced state. Consequently, we will obtain high-resolution structural information for both oxidation states.

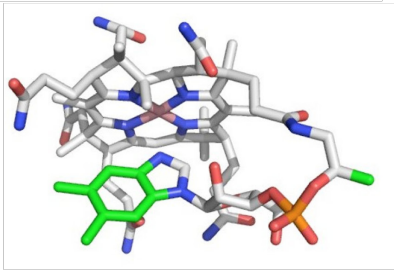
[1] O. Spiegelhauer *et al.* (2010). *J. Mol. Biol.* **396**, 66-82.
[2] J. Griese *et al.* (2006). *J. Mol. Biol.* **361**, 140-152.
[3] F. Spiegelhauer *et al.* (2009). *Biochemistry* **48**, 11412-11420.

Biological Degradation of Halogenated Organic Pollutants: Enzymes, Structures & Mechanisms

Martin Bommer, Holger Dobbek (Humboldt Universität zu Berlin)

The largest group of priority “persistent organic pollutants” identified by the United Nations Environment Program is the one comprising the class of halogenated, and in particular chlorinated, organic compounds. These are produced globally at industrial scale and are persistent in the soil at contaminated industrial sites. In addition, a number of undesirable compounds are also produced biologically. Chloromethane is mainly produced naturally and is a major contributor to ozone layer depletion. Similarly, most of the above-mentioned industrial pollutants also exist in the natural environment, albeit in much lower quantities. Conversely, the world's finely balanced ecosystem also provides a degradation route for these chemicals.





Top: *The Buna Chemical works at the Saale River in 1990, which provide a model system for dehalogenation*
Centre: *selected organic pollutants and their original applications*
Bottom: *An atomic model of Vitamin B12, which forms the core of the dehalogenase enzymes*

Within the BESSY-MX team, we are studying the dehalogenation reactions, which yield the harmless halide salt and non-chlorinated hydrocarbon. In nature, these are one step in complex enzymatic reaction cascades that provide energy for soil bacterial (dehalorespiration).

We are looking at the single enzyme (the biological catalyst) and its reaction mechanisms at atomic scale. Two of these are reductive dehalogenase, which dechlorinates the chemicals shown on the right, and chloromethane dehalogenase. Both enzymes use vitamin B12 (bottom) as co-factor, a complex corrinoid ring with a central redox-active cobalt ion (pink in the structure).

We hope to complete this picture and include the surrounding amino acid scaffold provided by the enzyme, which shields the activated cobalt and gives this reaction its specificity.

This will be investigated by X-ray diffraction using the synchrotron radiation at BESSY II and by time-resolved biochemical characterization at the Laboratory of Prof. Holger Dobbek at the Humboldt Universität zu Berlin.

[1] Hollinger *et al.* (1999) *FEMS Reviews* **22**, 383-398
[2] Futagami *et al.* (2008). *The Chemical Record* **8**, 1-12.

Biophysics

Thomas Hauß

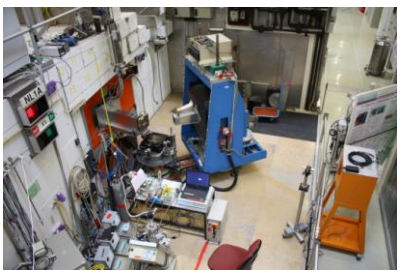
The biophysics group provides expertise on the investigation of proteins and especially biological membranes with neutron scattering techniques to study the structure and dynamics of these systems. The research topics include the investigation of biological (model) membranes with embedded peptides and proteins and proteins adsorbed to colloidal nano-particles.

Our group runs the neutron diffractometer V1 (Membrane Diffractometer) and the biophysics user laboratory (BioLab) on the Lise-Meitner-Campus Wannsee. The BioLab provides state-of-the-art instrumentation and techniques for guest scientists preparing and complementing their neutron scattering experiments on-site with a variety of biophysical and biochemical methods. One aspect of our work is to develop and provide unique and specialised sample environment, one example is a dedicated excess water cell to study membrane structures near physiological conditions. The Membrane Diffractometer served 10 external user experiments in 2010, 4 in-house experiments and 2 investigations with our long-term cooperation partner N.A. Dencher from the Technische Universität Darmstadt.

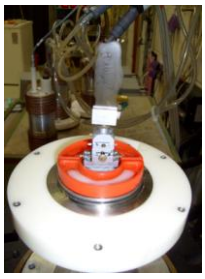
The main topics of the in-house research of our biophysics group are membrane biophysics and protein dynamics. With our longstanding expertise in this field, we are developing new techniques to study these systems by neutron diffraction, reflectometry and small angle scattering (both neutron and X-ray) very close to physiological conditions. The use of different scattering methods highlights specific characteristics of the systems under investigation.

The emerging trend for the explanation of neuro-degeneration in Alzheimer's disease imputes the cause of neurotoxicity to the interaction of soluble forms of amyloid-β peptide (Aβ) with neural cells and cell membranes. In a series of neutron diffraction and neutron small angle experiments we established that the toxic fragment Aβ(25-35) is able to penetrate and perturb lipid membranes and that the Aβ peptide induces membrane fusion. With quasi-elastic neutron scattering we observed an accelerated lateral diffusion of lipids in membranes doped with Aβ(25-35).

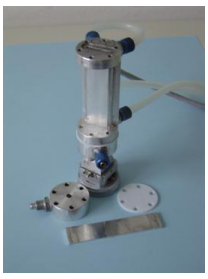
Very recently we developed in cooperation with J. Pieper (University of Tartu, Estonia) a new method to study in situ time resolved protein dynamics with a novel laser-pump – neutron-probe experiment. Our findings showed for the first time a modulation of the protein dynamics during a working cycle of a protein, here bacteriorhodopsin. Our goal is to characterise this modulation in dependence of important environmental parameters like hydration, temperature, pH, lipid composition, and others. To adopt this new method to proteins, which are not directly activated by a photon, the use of caged compounds, such as caged Ca, H+, or ATP in a flow-cell will be investigated.



Membrane Diffractometer V1



Sample mounted on V1



Excess water cell

Another area of interest in the biophysics group is the understanding of the mechanism of protein adsorption onto nanoparticles, as elucidated by their thermodynamic parameters. Protein adsorption onto nanomaterials is of interest in diverse applications including nanomedicines, food and waste processing, water purification and diagnostics. Isothermal titration calorimetry is used to determine the protein binding isotherm with varying temperature, salt and pH. The resulting changes in entropy, enthalpy and equilibrium constant can provide information about the relative importance of electrostatic and hydrophobic contributions to adsorption.

List of co-workers:

Dr. Thomas Hauß
Dr. Alexei Plotnikov
Hristina Karastaneva
Nicole Welsch
Alexandra Graebert
Luigi Sparacio

Selected publications 2009-2011:

1) Welsch N, Wittemann A, Ballauff M: *Enhanced activity of enzymes immobilized in thermoresponsive core-shell microgels*. J Phys Chem B 2009, **113**, 16039-16045.
2) Buchsteiner A, Hauß T, Dante S, Dencher NA: *Alzheimer's disease amyloid-beta peptide analogue alters the ps-dynamics of phospholipid membranes*. Biochim Biophys Acta 2010, **1798**, 1969-1976.
3) Ryabova NY, Kiselev MA, Dante S, Hauß T, Balagurov AM: *Investigation of stratum corneum lipid model membranes with free fatty acid composition by neutrondiffraction*. Eur Biophys J 2010, **39**, 1167-1176.
4) Welsch N, Ballauff M, Lu Y: *Microgels as Nanoreactors*. Applications in Catalysis 2010, **234**, 129-163.
5) Graebert A, Schmidt D, Kyriakopoulos A: *Selenoproteome of the nuclear envelope of JTC‑15 cells*. International Journal of Trends in Medicine 2011:*accepted*.
6) Wagner CS, Shehata S, Henzler K, Yuan J, Wittemann A: *Towards nanoscale-composite particles of dual complexity.* Journal of Colloid and Interface Science 2011, **355**, 115-123.
7) Dante S, Hauß T, Steitz R, Canale C, Dencher NA. *Nanoscale structural and mechanical effects of beta-amyloid (1-42) on polymer cushioned membranes: A combined study by neutron reflectometry and AFM Force Spectroscopy.* Biochim Biophys Acta. 2011 **Jul 24**. [Epub ahead of print]
8) Engelbrecht T, Schroeter A, Hauß T, Neubert RH. *Lipophilic penetration enhancers and their impact to the bilayer structure of stratum corneum lipid model membranes: Neutron diffraction studies based on the example Oleic Acid. Biochim* Biophys Acta. 2011 **Aug 18**. [Epub ahead of print]

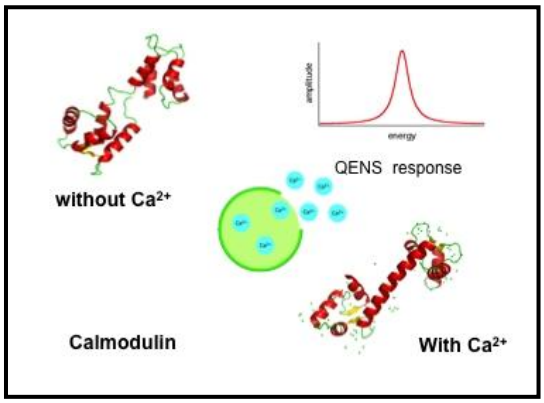
Time Resolved Protein Dynamics

Björn Drobot*, Thomas Hauß

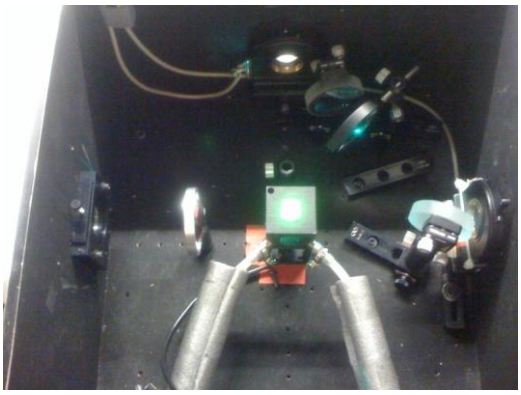
Proper functioning of a protein requires a well-defined three-dimensional structure. However, a protein is not a static entity; the working protein often undergoes larger conformational changes within microseconds and usually it needs a certain internal flexibility, which is provided by stochastic structural fluctuations on the picoseconds time scale. We have developed in cooperation with J. Pieper (University of Tartu, Estonia) a novel laser-pump – neutron-probe experiment, which combines in-situ optical activation of the function of a membrane protein, here bacteriorhodopsin, with a time-dependent sampling of the modulation of its protein dynamics using quasi-elastic neutron scattering [1,2].

To apply this technique to a more general class of proteins, we started to investigate the structural kinetics of calmodulin, a Ca^{2+} signalling amplifier. The idea is to study the dynamics during Ca^{2+} uptake or release in which the protein exhibits large structural changes.

To check the feasibility of calmodulin as a suitable system in terms of the kinetic response to a calcium pulse we started to investigate the time scale of the structural change. Here, we employ time-resolved fluorescence spectroscopy in a time domain from 5 ns to 1 s with intrinsic tryrosine fluorescence and specifically labelled calmodulin mutants. To realise this, calmodulin wild type and a single cystein mutant (CaM C75) was expressed in *E.coli* and purified. The calcium trigger is delivered by a caged Ca^{2+} compound (DM-Nitophen), which is activated by a short UV laser flash. The fluorescent light is recorded with a fast photomultiplier and a transient recorder with ns time resolution.



Structural change in Calmodulin,top left: apo-CaM, Bottom right: Ca^{2+} saturated CaM. The center symbolised the laser triggerd Ca^{2+} release. The inset top right depicts the future neutron scattering experiment, where we expect a difference in the quasi-elastic scattering during the large conformational change.



Sample chamber of the laser flash instrument. The laser acting light comes through the shutter at the top of the picture and is directed to the sample in the center. The excitation light is directed with 2 mirrors in a manner that it hits the sample quasi parallel to the acting light. The fluorescent light is collected by a lense and focussed to the entrance slit of a monochromator.

[1] Pieper J, Buchsteiner A, Dencher NA, Lechner RE, Hauß T, *Transient protein softening during the working cycle of a molecular machine.* Physical Review Letters 2008, **100**,228103.

[2] Pieper J, Buchsteiner A, Dencher NA, Lechner RE, Hauß T, *Light-induced modulation of protein dynamics during the photocycle of bacteriorhodopsin.* Photochem Photobiol 2009, **85**,590-597.

* Humboldt-Universität Berlin

Interactions between Proteins and Colloidal Particles

Nicole Welsch

The interface of materials science and biology is emerging as a major research focus, in particular, nanomaterials in medicine and biotechnology are expected to address many medical and biological problems. In these complex environments, nanoparticles come into contact with proteins, which can be adsorbed onto the particle surface. This changes the surface chemistry of the particle, and can also change the activity and conformation of the protein.

We investigate protein adsorption onto nanomaterials using two types of model nanoparticles. Spherical polyelectrolyte brushes (SPB) synthesized at the HZB consist of a solid polystyrene (PS) core onto which monodisperse polyelectrolyte chains are attached. Therefore, the adsorption of proteins on SPBs is electrostatically controlled. The second type of particles are core-shell microgels based on poly(N-isopropylacrylamide) (PNiPA). Herein, the PS cores are surrounded by a crosslinked PNiPA shell, which can be copolymerized with charged comonomers to produce microgels of different charged states. PNiPA exhibits a lower critical solution temperature (LCST) close to body temperature, where the microgel network shrinks and swells upon temperature changes (Fig. 1). Therefore, these smart core-shell microgels have the potential to adsorb and release proteins in a controlled way.

Calorimetric and scattering methods (e.g. Isothermal Titration Calorimetry, ITC and Small Angle X-ray Scattering, SAXS) are used to characterize the thermodynamics of the spontaneous adsorption process, as well as the spatial distribution of the proteins within the SPBs and microgels (Fig. 2).

In addition, kinetic experiments with immobilized enzymes are performed to determine the impact of immobilization on the enzymatic activity (Fig. 3). In all cases investigated the activity of adsorbed enzymes is retained or even increased upon adsorption. Furthermore, temperature-dependent kinetic experiments indicate that the catalytic activity of enzymes immobilized in thermosensitive microgels can be altered by the volume phase transition of the smart carriers.

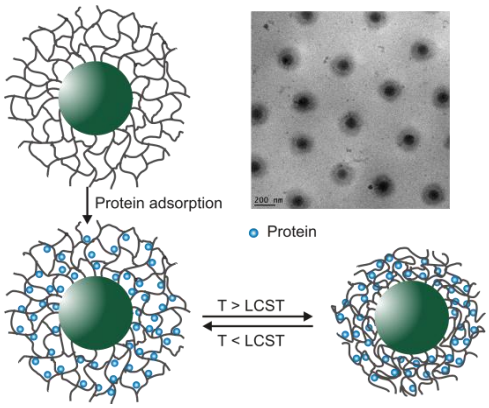


Figure 1: Top left and bottom: Schematic representation of the adsorption of proteins on PS-PNiPA microgel particles. Top right: Cryo-TEM image of these particles in the swollen state.

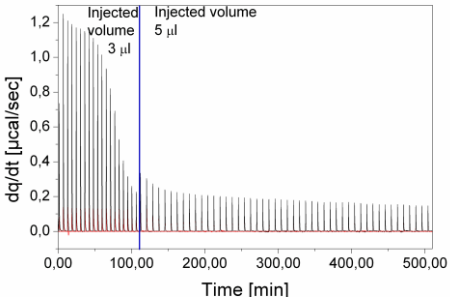


Figure 2: ITC data for titration of lysozyme into a solution of negatively charged microgels at pH 7.2 and at 25°C.

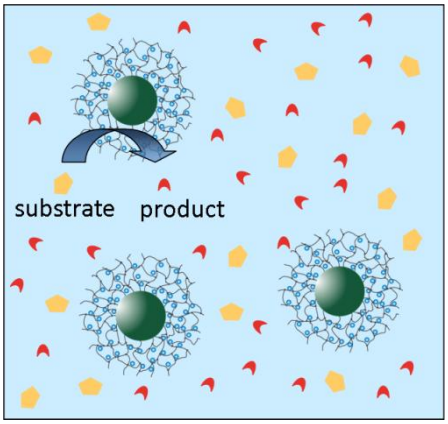


Figure 3: Schematic represent-tation of the catalysis with immobilized enzymes.

[1] Henzler, K. *et al*, Phys. Rev. Lett., 2008, **100**, 158301
[2] Henzler, K. *et al*, J. Am. Chem Soc., 2010, **132**, 3159-3163
[3] Welsch, N. *et al*, J. Phys. Chem. B, 2009, **49**, 16039-16045

Dynamics of Phospholipid Membranes Influenced by the Alzheimer's Disease Amyloid-β Peptide Analogue

Thomas Hauß

We investigate the influence of the neurotoxic Alzheimer's disease peptide amyloid-β and various fragments of it on the dynamics of phospholipid membranes by means of quasi-elastic neutron scattering (QENS) in the picosecond time-scale.

Samples of pure phospholipids (DMPC/DMPS) and samples with amyloid-β (25-35) peptide included have already been compared. With two different orientations of the samples the directional dependence of the dynamics was probed. The sample temperature was varied between 290 K and 320 K to cover both the gel phase and the liquid-crystalline phase of the lipid membranes.

The model for describing the dynamics combines a long-range translational diffusion of the lipid molecules and a spatially restricted diffusive motion.

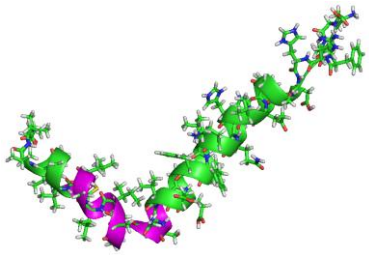
Amyloid-β (25-35) peptide affects significantly the ps-dynamics of oriented lipid membranes in different ways.

It increases the lateral diffusion velocity especially in the liquid-crystalline phase. This is very important for all kinds of protein-protein interactions which are enabled and strongly influenced by the lateral diffusion such as signal and energy transducing cascades. Amyloid-β (25-35) peptide also increases the local lipid mobility as probed by variations of the vibrational motions with a larger effect in the out-of-plane direction.

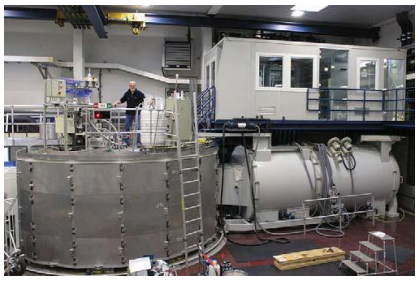
Thus, the insertion of amyloid-β (25-35) peptide changes not only the structure of phospholipid membranes as previously demonstrated by us employing neutron diffraction but also the dynamics inside the membranes.

The amyloid-β (25-35) peptide induced membrane alteration even at only 3 mol% might be involved in the pathology of Alzheimer's disease as well as be a clue in early diagnosis and therapy.

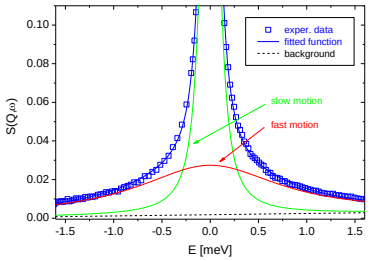
In future, we plan to investigate longer fragments of amyloid-β and its influence on natural membranes rather than phospholipid model membranes.



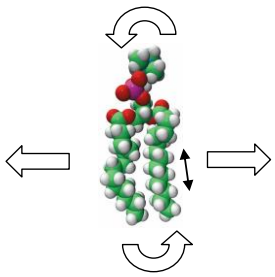
structure of amyloid-β (1-42) in aequeous solution (PDB ID: 1IYT) with the neurotoxic fragment amyloid-β (25-35) highlighted



time-of-flight spectrometer NEAT



typical QENS spectrum with two different motions



example of a phospholipid molecule with possible modes of motion indicated

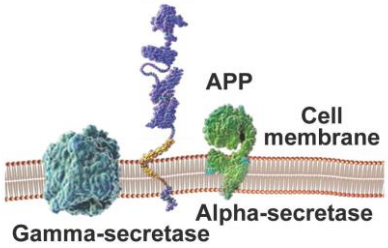
[1] A. Buchsteiner, T. Hauß, S. Dante, N.A. Dencher: *Alzheimer's disease amyloid-β peptide analogue alters the ps-dynamics of phospholipid membranes*, Biochimica et Biophysica Acta 2010, **1798**, 1969-1976

Interaction of a γ-Secretase Modulator with Model Lipid Membranes

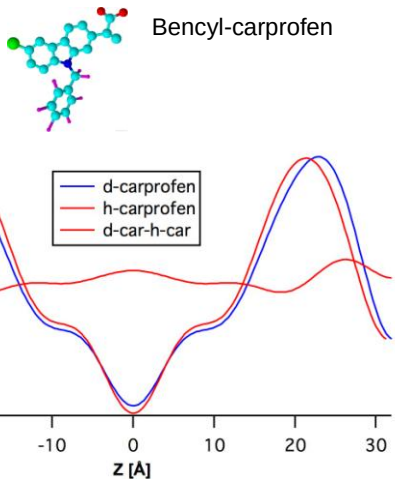
Thomas Hauß[1], Boris Schmidt[2], Norbert A. Dencher[3]

Recently, new strategies where developed to find a therapeutic approach to Alzheimer's disease [1,2,3]. The targets of the new approaches are enzymes responsible for the cleavage or modification of the amyloid precursor protein (APP) into the neurotoxic peptides β-amyloid with 40 to 43 amino acids. In the group of Professor Schmidt, TU-Darmstadt newly designed inhibitors and modulators for β-and γ-secretase were successfully tested in cultured cells and in mice [1,2]. The cleavage site of γ-secretase is in the hydrophobic core of the cell membrane, for that reason the inhibitor of γ-secretase is lipophilic.

We investigated by neutron diffraction the interaction of a newly synthesised and specifically deuterated γ-secretase modulator, a carprofen derivative, with lipid membranes. We established a suitable protocol for the preparation of biological highly relevant membrane models consisting of POPC, sphingomyelin, and cholesterol. This lipid mixture exhibits a change in lattice spacing, indicating a phase transition, at temperatures between 10°C and 40°C, with and without the inhibitor. The neutron diffraction experiments revealed the localization of the deuterated inhibitor in the membrane lipids as difference in the scattering length density profiles. The difference is calculated from samples of membrane lipids mixed with the protonated or selectively deuterated inhibitor carprofen, respectively, at two different contrast points (8% D2O, 20% D2O in the aqueous atmosphere) at 15°C. The maxima in the difference density profile at z = +- 2.5 nm are attributed to the location of the deuterated label. The tentative interpretation is, that the modulator with its deuterated bencyl ring resides in the head-group region of the lipid membrane close to the phosphate group of the lipids.



APP, alpha- and gamma-secretase embedded in a cell membrane



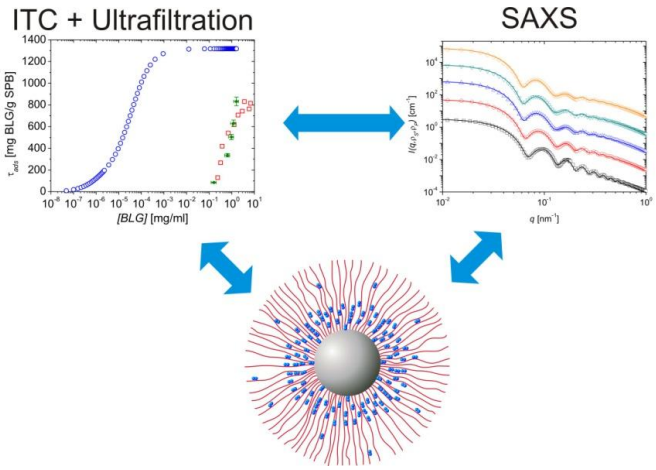
Neutron scattering length density profiles of lipid membranes with deuterated and protonated carprofen derivatives, respectively, and its difference indicating the position of the deuterated bencyl ring

[1] Rajendran et al. *Efficient inhibition of the Alzheimer's Disease β-seccretase by Membrane targeting*, Science **320**, 520 (2008)
[2] Kukar et al., *Substrate-targeting γ-secretase modulators*, Nature **453**, 925 (2008)
[3] Schilling et al., *Glutaminyl cyclase inhibition attenuates pyroglutamate A and Alzheimer's disease–like pathology,* Nature Medicine **14**, 1106 (2008)

1) Helmholtz-Zentrum Berlin, Institut für weiche Materie und funktionale Materialien
2) Technische Universität Darmstadt, Clemens Schöpf-Institut, Organische Chemie,
3) Technische Universität Darmstadt, Clemens Schöpf-Institut, Physikalische Biochemie

Interaction Strength between Proteins and Polyelectrolyte Brushes: A Small Angle X-Ray Scattering Study

K. Henzler[1,2], B. Haupt[1,2], S. Rosenfeldt[2], L. Harnau[3], T. Narayanan[4], M. Ballauff [1] “



The interaction of proteins with different surfaces is of central interest for biotechnology and medical science. Here, we present an investigation of the adsorption of β-lactoglobulin (BLG) onto spherical polyelectrolyte brushes (SPB) by small angle X-ray scattering, isothermal titration calorimetry and extensive ultrafiltration. This demonstrates for the first time that an in-depth understanding of the interaction strength of proteins with polyelectrolyte brushes can be obtained by the proper combination of different analytical methods. The amount and distribution of the protein insight the brush layer can be determined by small angle X-ray scattering (SAXS).[1] Furthermore, the SAXS measurement shows that a certain amount of the protein molecules form linear aggregates in the adsorbed state of about six monomer units. On the other hand, isothermal titration calorimetry (ITC) provides the opportunity to investigate the thermodynamics of the protein adsorption as well as the amount of adsorbed protein at the equilibrium.[2] The amount of adsorbed BLG determined by ITC and SAXS can be compared. The third method used in this investigation is the extensive ultrafiltration.[3] By this method the amount of tightly bound BLG can be determined. From the SAXS analysis the amount of adsorbed protein in different parts of the polyelectrolyte layer can be calculated. These results can be compared to the data obtained by ultrafiltration. It is found that the proteins which are bound in the outer part of the brush layer can be washed out by ultrafiltration.

[1] Henzler et al., *Directed motion of proteins along tethered polyelectrolytes*, Phys. Rev. Lett. **100**, 158301 (2008)

[2] Henzler et al., *Adsorption of beta-Lactoglobulin on Spherical Polyelectrolyte Brushes: Direct Proof of Counterion Release by Isothermal Titration Calorimetry*, J. Am. Chem. Soc. **132**, 3159 (2010)

[3] Wittemann et al., *Interaction of proteins with linear polyelectrolytes and spherical polyelectrolyte brushes in aqueous solution*, Phys. Chem. Chem. Phys. **8**, 5269 (2006)

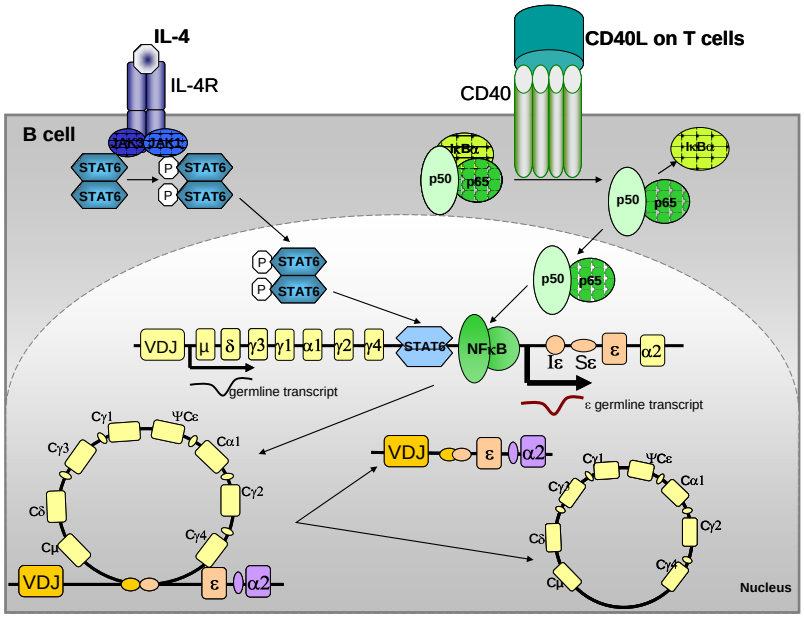
[4] Henzler et al., *Interaction strength between proteins and polyelectrolyte brushes: A small angle X-ray scattering study*. Phys. Chem. Chem. Phys. 2010, *accepted*

1) Helmholtz-Zentrum Berlin, Institut für weiche Materie und funktionale Materialien
2) Universität Bayreuth, Physikalische Chemie I
3) Max-Planck-Institut für Metallforschung Stuttgart; Universität Stuttgart, Institut für Theoretische und Angewandte Physik
4) European Synchrotron Radiation Facility Grenoble

Intracellular localization of docosahexaenoic acid (DHA) in human B cells

Christin Weise*, Alexandra Graebert, Margitta Worm*

The changing polyunsaturated fatty acid (PUFA) proportion of westernized nutrition with a decrease in omega-3 (n-3) species may contribute to the globally growing incidence of allergic diseases, like atopic eczema, allergic rhinitis and asthma. Evidences from *in vitro, ex vivo* and animal studies support the immunomodulatory effects of dietary long-chain n-3 PUFA known from fish oil. According to the position of its first double-bound counted from the acyl terminus, DHA (C22:6n-3) belongs to this group of PUFA which have been suggested to be candidates for dietary intervention of allergic diseases. Our previous clinical results imply that DHA supplementation improves slightly the clinical outcome of atopic eczema and results in a reduced ex vivo production of IgE, the key effector molecule of Type I allergies.[1] Investigation of the underlying molecular mechanisms revealed that DHA leads to a profound repression of the IgE switching process, IgE plasma cell development and IgE production in human B cells through early inhibition of CD40 and IL-4 signaling pathways.[2]



IgE production requires class switch recombination which is regulated at the level of germline transcription (GLT) of constant heavy chain genes and induction of activation induced desaminase (AID) expression. Thereby, two signals induce the class switch to IgE. CD40 ligation leads to liberation of NFκB, which translocates into nucleus and regulates the expression of a plenty of genes. IL-4 binding to its receptor complex induces the nuclear translocation of STAT6 and thus the modulation of transcription of IL-4 responsive genes. NFκB and STAT6 bind to the ε-germline gene promoter initiating the transcription of the sterile εGLT. Association of εGLT with the ε-switch region marks the target DNA sequence for AID leading to the DNA rearrangement process. Both, NFκB and STAT6 synergize for IgE transcription and optimal IgE production.

Fatty acid organization into distinct cellular pools plays a particularly important role in immune cells. Forms of lipid trafficking exist, but are poorly understood. In contrast to saturated fatty acid there is a lack of data about uptake, transport, and intracellular distribution of very long chain fatty acids, especially of DHA. To show whether and to what extent subcellular compartments accumulate DHA in human B cells, lymphocytes will be subjected to radio-labeled DHA. Upon subcellular fractionation, DHA content of respective fractions will be determined by radiography.

[1] C. Koch *et al.*; *Br. J. Dermatol*, **2008**. *158*, 786. [2].C. Weise *et al.*, *J. Nutr. Biochem*, **2011**. *22*, 269.

* Immunomodulation group, Allergie-Centrum-Charité, Charité - Universitätsmedizin Berlin, Cooperation partner

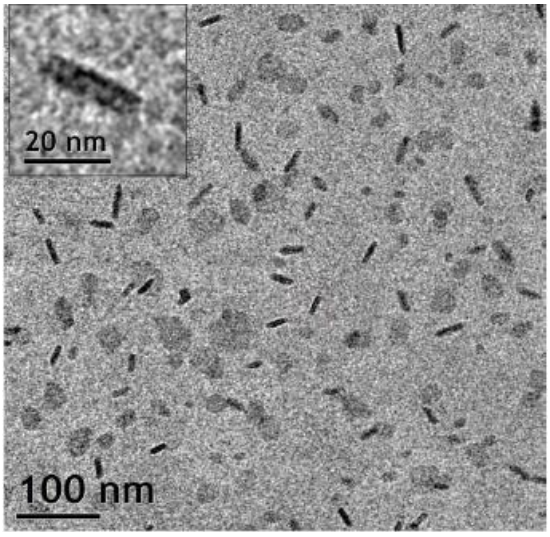
Colloid Physics

Guenter Goerigk, Daniel Clemens

The colloid physics group is providing instrumentation for investigations of structure and dynamics of large scale structures using neutrons, X-rays and light as suitable probes. We continue to develop our techniques in order to access important outer parameters as temperature, shear forces, etc. that have an influence on the systems under investigation. Our instrumentation includes small-angle scattering (V16/VSANS) and spin-echo spectroscopy (V5/SPAN) on the neutron side. We cooperate in the operation of an anomalous small-angle X-ray scattering (ASAXS) beam line at the BESSY II synchrotron with F-A2 (Microstructure and residual stress analysis).

The colloid physics group is also in charge of the colloid lab. Here we are running additional methods as e.g. static and dynamic light scattering, zeta-sizer as well as rheometers that supplement investigations by the beamlines of our users. These facilities are open to all users of the beamlines of F-I2. Additional techniques are assembled in the newly founded **Joint Laboratory of Structural Research** (JLSR) which is run in close collaboration with the Institute of Physics of the Humboldt University. A new cryogenic transmission electron microscope (cryo-TEM) started operation in October 2011. This instrument allows us to analyze suspensions of colloidal and biological systems in aqueous environment (Figure). The results obtained in this way can directly be compared to similar studied done with the X-ray microscope (see the description of the X-ray microscopy group). Moreover, the analysis done in real space is compared to small-angle experiments done by SANS and SAXS.

The group is engaged in selected research activities, e.g. in the field of applied small-angle scattering of colloids: microdomains in lipid membranes, critical phenomena at mesoscopic scale, comparison to theoretical predictions for polyelectrolyte brushes and the rheology of hard sphere suspensions. Moreover, we have an intense cooperation with research groups in the field of solar cells, plasmonics and catalysis within and outside HZB.



Cryo-TEM micrograph of an aqueous suspension of polyethylene nanocrystals. The inset displays an enlarged picture of one crystal. This analysis has been combined with SAXS to arrive at a full model of the smallest polymer crystals made so far. Taken from ref. [12] selected publications.

A point of special interest is the combination of cryo-TEM with scattering methods. Recently, we analyzed nano-crystals from polyethylene in collaboration with the group of Prof. S. Mecking, University of Konstanz. The shape and the size distribution of the particles was assessed directly by cryo-TEM while their internal structure was analyzed by SAXS including contrast variation. In this way a full structural model of these particles that present the smallest polymer crystals ever synthesized could be achieved. Moreover, the lamellar thickening of the nanocrystals after thermal annealing could be analyzed.

Coworkers:

Dr. Beate-Annette Brüning

Dr. Daniel Clemens

Dr. Günter Goerigk

Dr. Sylvain Prévost

Christian Rabe

Miriam Siebenbürger

Dr. Ralf Stehle

Dr. Karsten Vogtt

Selected Publications in 2009-2012:

1. M. Hoffmann, A. Jusufi, C. Schneider, M. Ballauff: *Surface potential of spherical polyelectrolyte brushes in the presence of trivalent counterions*; J. Colloid Interface Sci., **338**(2), 566-572 (2009).
2. S. Lyonnard, Q. Berrod, B. Brüning, G. Gebel, A. Guillermo, H. Ftouni, J. Ollivier, B. Frick: *Perfluorinated surfactants as model charged systems for understanding the effect of confinement on proton transport and water mobility in fuel cell membranes. A study by QENS*; Eur. Phys. J ST **189**, 205-216 (2010).
3. B. Brüning, M.C. Rheinstädter, A. Hiess, B. Weinhausen, T. Reusch, S. Aeffner, T. Salditt: *Influence of cholesterol on the collective dynamics of the phospholipid acyl chains in model membranes*; Eur. Phys. J E **31**, 419-428 (2010)
4. B. Brüning, E. Wald, W. Schrader, R. Behrends, U. Kaatze: *Slowing down in lipid bilayers: domain structure fluctuations and axial diffusion*; Soft Matter **5**, 3340 (2009).
5. F. Cousin, J. Gummel, D. Clemens, I. Grillo, F. Boué: *Multiple Scale Reorganization of Electrostatic Complexes of PolyStyreneSulfonate and Lysozyme*; Langmuir **26**, 7078–7085 (2010).
6. M. Hoffmann, M. Siebenbürger, L. Harnau, M. Hund, C. Hanske, Y. Lu, C.S. Wagner, M. Drechsler, M. Ballauff: *Thermoresponsive colloidal molecules*. Soft Matter **6**, 1125-1128 (2010).
7. H.H. Winter, M. Siebenbürger, D. Hajnal, O. Henrich, M. Fuchs, M. Ballauff: *An empirical constitutive law for concentrated colloidal suspensions in the approach of the glass transition*. Rheol. Acta **48 (7)**, 747-753 (2009).
8. M. Siebenbürger, M. Fuchs, H. Winter, M. Ballauff: *Viscoelasticity and shear flow of concentrated, noncrystallizing colloidal suspensions: Comparison with mode-coupling theory*. J. Rheol. **53(3)**, 707-726 (2009).
9. K. Vogtt, C. Jeworrek, V. Garamus, R. Winter: *Microdomain Formation in Lipid Vesicles: Structure and Distribution Assessed by Small Angle Neutron Scattering,* Journal of Physical Chemistry B **114**(16), 5643–5648 (2010).
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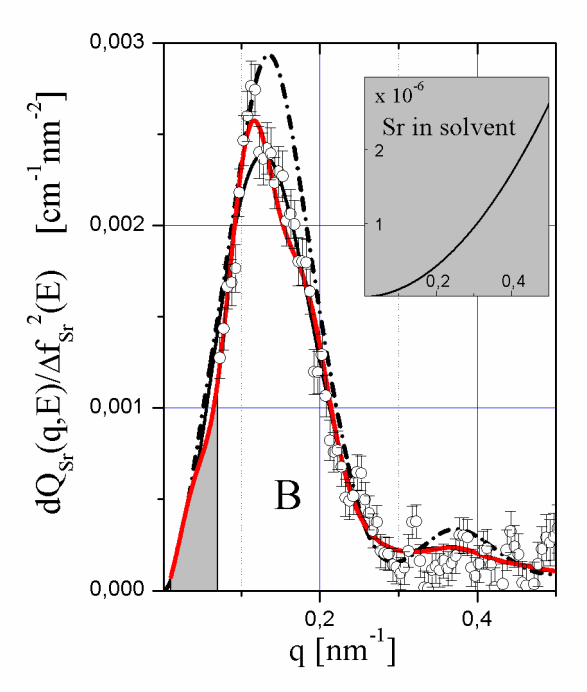
13. V. Mengarelli, M. Zeghal, L. Auvray, D. Clemens: *Phase behavior and structure of stable complexes between a long polyanion and a branched polycation;* Physical Review E **84**, 1-12 (2011)
14. M. Siebenbürger, M. Fuchs, M. Ballauff: *Core-shell microgels as model colloids for rheological studies*, Soft Matter 2012, DOI: 10.1039/C2SM07011A.

Anomalous Small-Angle X-ray Scattering – A Precise Experimental Technique for the Quantitative Analysis of Nano-scaled Phases in Chemistry and Physics

Günter Goerigk



Small-Angle Scattering (SAS) experiments average over a large sample volume and give structural and quantitative information of high statistical significance on a mesoscopic length scale between 1 and hundreds of nanometers, which can be correlated with macroscopic physical and chemical parameters of the analyzed materials. The materials under investigation cover a wide range of different scientific fields in chemistry, solid state physics, catalysis research, material and membrane science. Examples are macromolecules in solution, suspensions, metal nanoparticles on porous support structures, composites, membranes, alloys, semiconductors, glasses...). Synchrotron radiation (SR) provides extraordinary powerful tools for SAS experiments.

Anomalous Small-Angle X-ray Scattering (ASAXS) with synchrotron radiation employs the energy dependence of the atomic scattering factors in the vicinity of the K- and L_{III}-absorption edges of most of the elements giving access to the element-specific structural and quantitative characterization of the samples under investigation. From a series of publications of the last years it has been shown, that tremendous quantitative information about chemical concentrations in highly diluted chemical solutions can be obtained by *q*-ASAXS when employing the so-called Resonant Invariant (RI) [1-3]. From the integral (RI) in Figure 1 the amount of Sr counter ions localized in collapsed subdomains of polyacrylate with respect to the total amount of Sr-cations in the solvent was deduced, while approaching the systems phase boundary. From the distinct dependence of the ratio r=[Sr^{2+}]/[PAA] deduced from the concentrations of Sr^{2+} in the condensed phase a higher Sr^{2+} binding was confirmed when approaching the phase boundary, which had been previously established by light scattering [4]. The example demonstrates the capability to determine quantitatively the extent of ion binding to polyelectrolytes which condense to or interact specifically with the polyelectrolyte.

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[4] Schweins R, Goerigk G, Huber K, *Eur. Phys. J. E* **2006**; 21: 99

Analysis of the Critical Casimir Effect in Binary Liquid Mixtures by V-SANS

Günter Goerigk, Miriam Siebenbürger, Nico Grimm, Yan Lu, Dirk Wallacher, Katja Henzler, Christian Rabe, Matthias Ballauff

If a fluctuating medium is confined, Casimir-like forces acting on the confining surfaces are generated. Near the continuous phase transition of such a medium the corresponding order parameters cover all length scales and in the neighbourhood to the critical point universality occurs i.e. the critical phenomena become to a large extent independent of the microscopic (atomistic) details of the system under investigation. Recently theoretical and experimental results have been reported on the direct measurement of the critical Casimir forces by total internal reflection microscopy [1]. The corresponding potentials have been determined for individual colloidal particles floating above a substrate under the action of the critical thermal noise in the solvent medium, constituted by a binary liquid mixture of water and lutidine near the critical point. Depending on the properties of the (large) colloids and substrate surfaces attractive respectively repulsive forces have been observed, when approaching the critical point of the mixture and quantitative parameters related to the effective potential were deduced.

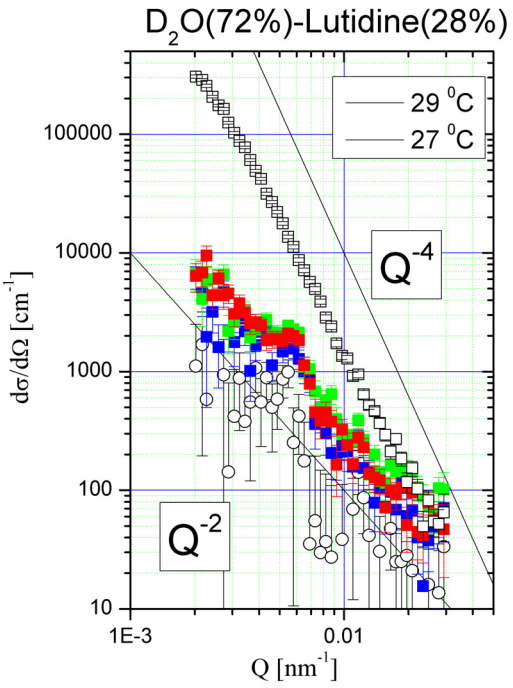

Figure 1: V-SANS curves of a critical D_2O(72%)/2.6-Lutidine(28%) mixture for selected temperatures between 27 and 29 ^{0}C (T_C for H_2O at 28.85 ^{0}C). Hollow squares $T > T_C$.

Due to the related colloid sizes in the micrometer range these experiments are well suited for V-SANS measurements in transmission geometry especially for the KWS-3@FRM II [2]. For this purpose a sample cell was developed, which meets the special requirements of the neutron beam at KWS-3 (special sapphire windows with suitable size), providing a temperature stability of better 5 mK at RT. 1st measurements with this prototype have been performed in December 2011. Figure 1 summarizes the V-SANS curves of a critical D_2O(72%)/2.6-Lutidine(28%) mixture for selected temperatures between 27 and 29 °C (TC for H_2O at 28.85 °C). The four scattering curves with black, blue, green and red symbols show the typical asymptotic behaviour of concentration fluctuations with a Q^{-2} dependence (Ornstein-Zernike) while the black square symbols represent the scattering of a two phase mixture beyond the critical temperature (at 29 °C) with an asymptotic Q^{-4} dependence (Porod).

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Correlation of Nanostructure and Photoconductivity in Amorphous Silicon-Germanium Alloys – Solar Cell Materials Analyzed by Anomalous Small-Angle X-ray Scattering

Günter Goerigk

Hydrogenated amorphous silicon-germanium alloys are used in solar cell technology, where the germanium is added to produce lower band gap material to absorb the longer wavelength photons of the solar spectrum and to achieve higher efficiences. Previous small-angle X-ray scattering (SAXS) and anomalous small-angle X-ray scattering (ASAXS) studies revealed that, in addition to voids, non-uniformly distributed Ge contributes to the material inhomogeneities, which are strongly related to the degradation of the opto-electronic properties. In recent years there has been a growinginterest especially in the hot-wire chemical-vapor depostion (HWCVD) technique due to evidence of improved stability and improved opto-electronic properties of the material, as well as the potentially beneficial manufacturing feature of higher deposition rates than the current industrial technique of plasma-enhanced chemical vapor deposition (PECVD). A group at NREL (National Renewable Energy Laboratory, U.S.A.) found evidence of improved photoresponse from HWCVD a-SiGe:H alloys with narrow bandgaps deposited at different filament temperatures, filament diameters, and optimized substrate temperatures [1,2].

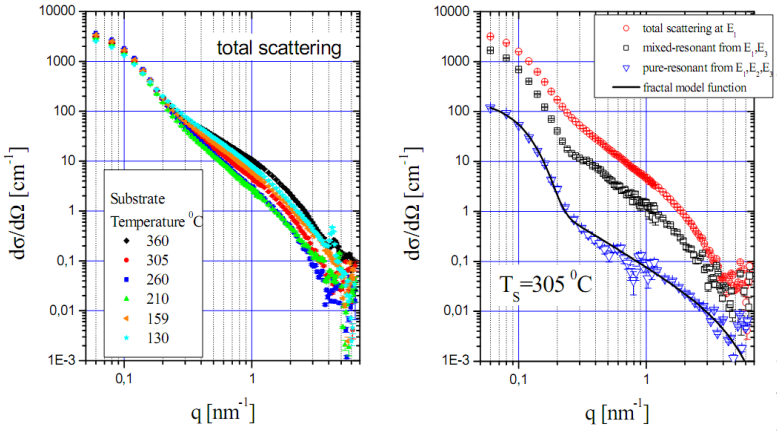

Fig. 1: SAXS curves of hotwire deposited amorphous Si-Ge alloys used in solar cell techniques. In the left picture strong changes due to the variation of the substrate temperature occur. In the right picture results from an ASAXS measurements of one sample per-formed near the Ge-K-edge (11.103 eV) are depicted. The blue triangles represent the nanostructure of only the Ge-contain-ing component. The solid line through the blue triangles represents the scattering of mass fractals with dimension 1.6 and a size of 40 nm. Model function taken from [8].

The nanostructure of hydrogenated amorphous silicon-germanium alloys, a-$Si_{1-x}Ge_x$:H (over a wide range in x), prepared by PECVD and the hot-wire deposition technique applying different substrate and filament temperatures have been analyzed in a series of publications in the last decade by anomalous small angle x-ray scattering experiments [3-7]. For all alloys the Ge-component was found to be inhomogeneously distributed. The results from the structural and quantitative analysis have been correlated to the material photoconductivity. A clear improvement of the photoconductivity was achieved by optimizing the substrate temperature of the hotwire deposited films (between 130 and 360 ºC) due to the reduction of hydrogen containing voids in coincidence with the formation of mass fractal structures of Ge with the fractal dimension p < 1.6 and a size of about 40 nm. The two processes cause the structural re-organization of Hydrogen from voids into Ge-fractals with enhanced Ge-H bonding, thereby improving the material photoconductivity.

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Analysis of interaction of thermoresponsive colloidal dumbbells by V-SANS

Günter Goerigk, Katja Henzler, Martin Hoffmann, Miriam Siebenbürger, Christian Rabe, Matthias Ballauff

The term „colloidal atom" introduced by van Blaaderen describes the analogy between single atomic gases like helium and spherical colloidal particles [1]. Following this analogy, the increasing complexity of the model system leads to the hydrogen molecule with its colloidal equivalent, the dumbbell shaped particle. We synthesized colloidal particles with a dumbbell shape consisting of a poly(styrene)-poly(methylmethacrylate) core and a crosslinked thermoresponsive poly-(N-isopropylacrylamide) layer around the anisotropic core [2]. The size of these anisotropic particles is around 180 nm in the length and 90 nm in the width at room temperature in aqueous solution determined by DLS, DDLS and cryo-TEM. Due to the thermosensitivity both the volume fraction and the aspect ratio of the particles can be adjusted.

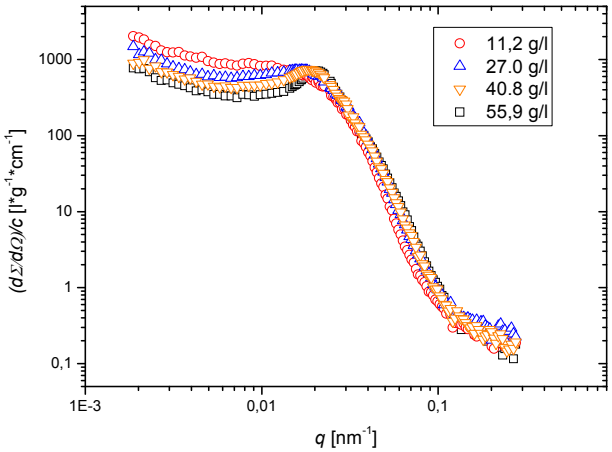

Fig 1: Scattering intensity of the dumbbell-shaped colloidal particles normalized to the particle concentration plotted versus the scattering vector Q. As solvent, a H_2O/D_2O mixture (43.21/56.79 Vol-%) without additional salt was chosen. The different symbols represent the different particle concentration in g/l. Here, a marked dependency of the structure factor on the particle concentration is found.

SANS and V-SANS are powerful tools to investigate the structure and the interaction of these particles in aqueous solution. Figure 1 shows the first scattering curves of dumbbell-shaped thermoresponsive particles in a H_2O/D_2O mixture (Vol-% ratio: H_2O/D_2O= 43.21/56.79) at different particle concentrations. The measurements were performed at the V-SANS instrument KWS-3@FRMII at two sample detector distances (9.3 m and 1.1m) covering more than two orders of magnitude in Q-range between 0.0018 nm^{-1} and 0.3 nm^{-1}. Here, it becomes obvious that the available Q-range at the KWS-3 is highly suited to investigate the structure factor and the overall size of these particles in aqueous solution [3]. These thermoresponsive particles provide the chance to adjust the volume fraction of the particles by increasing the particle concentration and by changing the system temperature. Thus, volume fractions up to 0.65 are achievable. Hence, an investigation of the glassy state is possible by scattering techniques. Moreover, the contrast variation method allows to investigate the dependence of the radius of gyration R_g on the solvent contrast [4]. Anisotropic particles should show a different behaviour compared to spherical particles with rotational symmetry.

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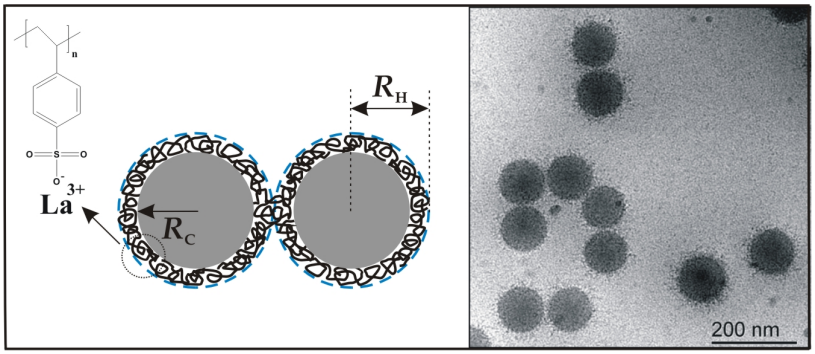
The Colloidal Stability of Spherical Polyelectrolyte Brushes

Christian Schneider

Polyelectrolyte brushes are systems in which long polyelectrolyte chains are densely attached to planar or curved surfaces. Attaching long polyelectrolyte chains to colloidal core particles leads to spherical polyelectrolyte brushes (SPBs).
Due to the very high concentration of ionic groups in the polyelectrolyte shell, almost all of the corresponding counter ions are confined to the shell. In the presence of monovalent counter ions, this leads to a high concentration of counterions inside the shell layer. As such, the osmotic pressure of these confined counter ions is very high, leading to a stretching of the polyelectrolyte shell layer. The stretched polyelectrolyte chains evoke the high stability of the SPBs against coagulation, due to both steric (stretching) and electrostatic (charges on the chains) interactions. Addition of traces of multivalent counter ions, however, first results in an ion exchange inside the shell layer, whereby few multivalent ions are getting trapped and many monovalent ions released. The ion exchange process is accompanied by a decreasing particle stability, as the shell layers of the SPBs collapse.



Schematic (left) and cryo-TEM (right) picture of collapsed and instable SPB particles.

These “electrosteric” stabilized core-shell colloidal systems are widely utilized in industrial applications. We have shown that SPBs serve as good model systems for “electrosteric” stabilized colloids. Thus, we investigate the stability behaviour of SPBs via simultaneous static and dynamic light scattering in the presence of multivalent counter ions.
In the frame of a NSF-DFG cooperation, we work closely with colleagues at the University of California Berkeley and Temple University. During this cooperation, we achieved two goals so far: First, we can now measure the surface potential of an anionic SPB system as a function of the multivalent La^{3+} counter ion concentration in the size order of the thermal energy. Second, given important system parameters of the SPB like contour length and the grafting density of the polyelectrolyte chains, we can predict the stability behaviour of the SPB system within a remarkably high accuracy. Our next steps aim at testing the validity of our model to a wide range of experimental parameters, like SPB-type, type of counter ion and counter ion valency.

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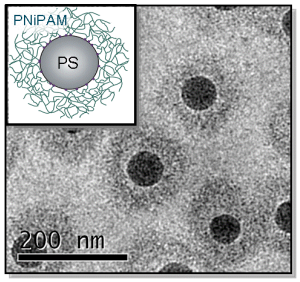
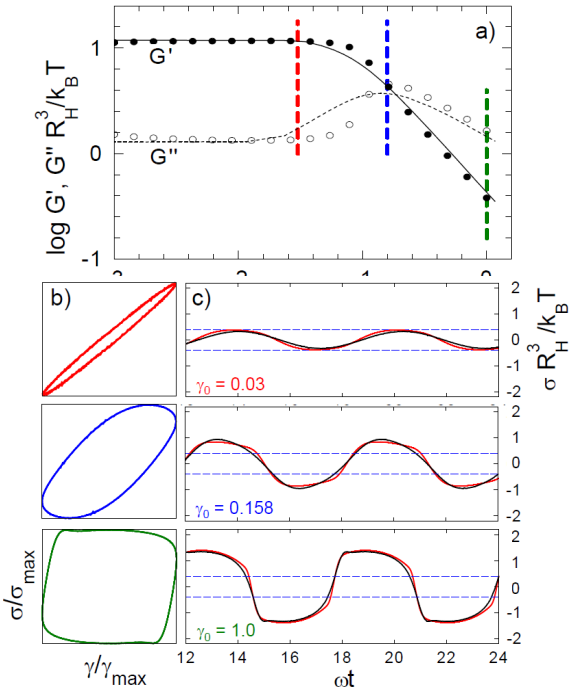
Yielding of a Concentrated Suspension Observed by FT-Rheology: Comparison with the Mode Coupling Theory

Miriam Siebenbürger, Joseph Brader*, Matthias Fuchs**



The dynamics and the mechanical properties of hard sphere fluids at high concentrations are an old, but nevertheless a very interesting topic in research. In particular, the transition from the fluid to the glassy state at a volume fraction of $\phi_g \approx 0.58$ and the glass itself above this volume fraction are not completely understood yet. As a model system to mimic the theoretical hard sphere fluid, aqueous suspensions of particles consisting of a poly(styrene) (PS) core and a thermo-sensitive poly(*N*-isopropylacrylamide) (PNiPAM) shell are used [1]. Suspensions in the fluid state at high concentrations (close to ϕ_g) show a shear thinning behaviour. In the glass a yield stress σ_y is found. The low force needed for shear melting colloidal glasses and crystals allows rheological investigations at volume fractions above ϕ_g.

The Mode Coupling Theory (MCT) quantitatively describes the dynamics and the mechanical properties of hard sphere suspensions for the fluid and the glassy state close to the glass transition in the stationary flow and the linear viscoelastic regime [2, 3]. The yielding process of the glass can be followed from the linear to the non-linear viscoelastic regime by an oscillatory deformation test as shown in a). The Fourier transformation rheology (FT-rheology) [4] is a quite new rheological technique, which allows the quantification of the degree of non-linearity by means of the intensity ratio of higher harmonics to the fundamental frequency. This quantity can be obtained from the sample by an oscillatory time test at one frequency and strain amplitudes γ_0 as shown in c) by a Fourier transformation. With the schematic MCT-model used to describe the steady state shear and the linear viscoelastic regime it is also possible to calculate the non-linear regime without further parameters (see a) and c)).



a) Comparison of the experimental deformation test (symbols) at 1Hz in the glassy state at ϕ_g = 0.65 with the MCT-calculations (black lines) [5]. At strains of γ_0 = 0.03 (red), γ_0 = 0.158 (blue) and γ_0 = 1.0 (green) time tests with the FT-rheology are performed. The results are given in b) and c).

b) Lissajous diagrams of the time tests. The enclosed area is directly correlated to the dissipated energy [5].

c) Time tests at 1Hz and the given strain amplitudes γ_0; experimental results are given as black lines, MCT-calculations are drawn in red. The blue dashed lines indicate the yield stress σ_y [5].

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* Université de Fribourg ** Universität Konstanz

V16 - A New SANS Instrument for Soft Matter Research

Karsten Vogtt, Daniel Clemens

Small angle scattering techniques detect the global structure of particles in solution in the range from a few to hundreds of nanometers. In particular small angle neutron scattering (SANS) is sensitive for the distribution of atom types rather than electron density or polarizability as it is the case for small x-ray and light scattering, respectively. It thus allows isotope labeling of samples and solution without disturbing the chemical nature of the sample and its environment. In this manner SANS allows additionally the characterization of the inner structure of particles *in situ*.

The new SANS instrument V16/VSANS is dedicated to use the benefits of this method and complements them with the so called “time-of-flight” option. This technique utilizes choppers to create distinct neutron pulses (see Fig. 1) in combination with a detector, which detects the position of incident neutrons and their time of arrival at the detector position. These gathered data yield position and wavelength of each detected neutron using a polychromatic neutron beam. Thus a broad – and via the chopper setup - tunable range of scattering momenta can be scanned in a single experiment. A further operational mode allows employing a highly focused beam employing a multi-pinhole grid, which enables the detection of very low scattering angles and hence the determination of particles with sizes up to nearly a micrometer. Installation and operation of VSANS is underpinned by the simulation of the full instrument using the ray-tracing software package VITESS, developed at HZB.

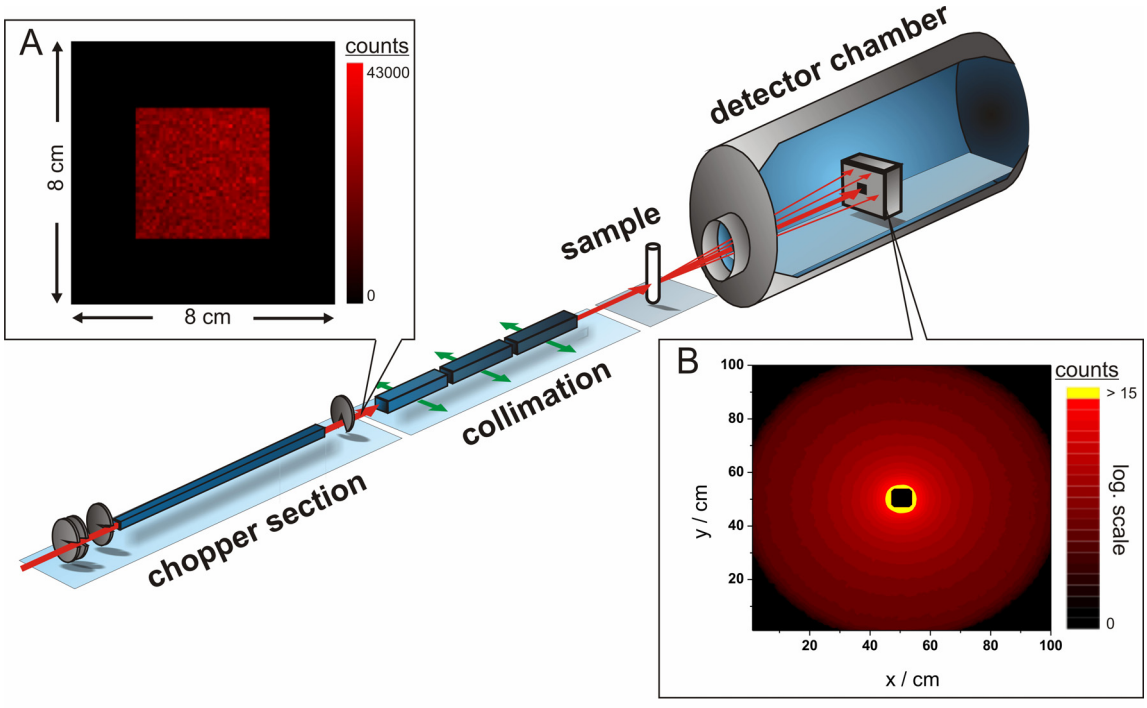

Figure 1: Isometric sketch of the instrument V16 from the chopper section (left) to the detector chamber (right, for a better vision on the detector a part of the chamber wall was virtually cut out). Inset A (upper left corner) depicts the distribution of neutrons over the neutron guide cross section behind chopper 4 taken from a VITESS-simulation. Inset B shows the according detector picture simulated for a sample of spherical particles (R = 54 nm).

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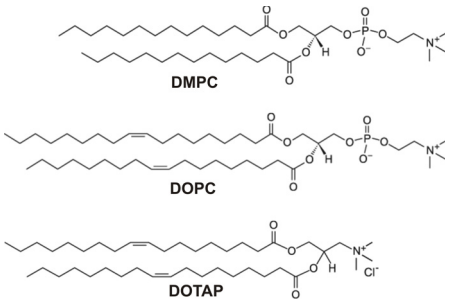
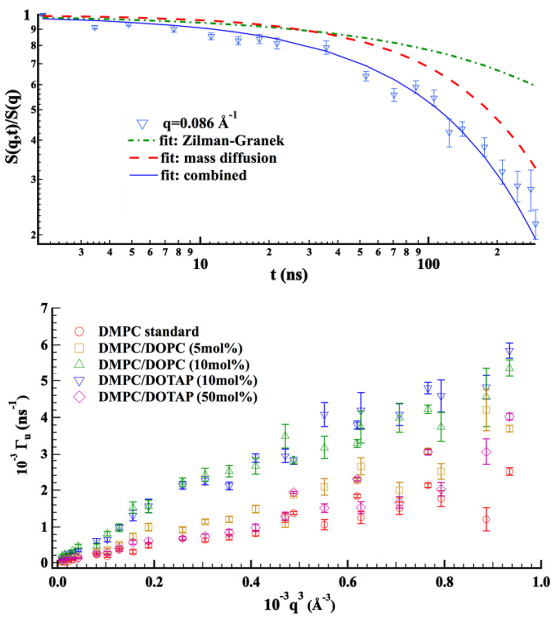
Influence of charge density on bilayer bending rigidity in lipid vesicles: a combined dynamic light scattering and neutron spin-echo study

Beate-Annette Brüning[1], Ralf Stehle[1,2], Peter Falus[3], Bela Farago[3]

Liposomes composed of cationic lipid mixtures have been found effective vehicles for cellular drug delivery [1], as well as gene transfection applications [2]. A detailed understanding of the changes induced in model vesicles by the insertion of charged lipids is crucial to exploit their potential as targeted carriers. We report a combined dynamic light scattering and neutron spin-echo study on vesicles composed of the uncharged helper lipid 1,2 dimyristoyl-sn-glycero-3-phosphatidylcholine (DMPC) and the cationic lipid 1,2-dioleoyl-3-trimethylammonium-propane (DOTAP). We compare the bilayer undulation dynamics in DMPC/DOTAP vesicles to those composed of a mixture of the uncharged helper lipid DMPC with the also uncharged reference lipid 1,2-dioleoyl-sn-glycero-3-phosphocholine (DOPC). We have performed dynamic light scattering on both types of lipid mixtures to investigate changes in vesicle size and mass diffusion. We study bilayer undulation and bulk diffusion dynamics using neutron spin-echo spectroscopy on two distinct time scales, namely around 25 ns and 150 ns. Finally, from combined fits including contributions of vesicle mass diffusion and bilayer undulation dynamics we calculate the respective bilayer bending rigidities κ_B for both types of lipid vesicles [3]. We find, that on the local length scale the insertion of lipid headgroup charge influences the bilayer undulation dynamics and bilayer bending rigidity κ_B less than inserting lipids with acyl chain unsaturation: We observe a bilayer softening with increasing inhomogenity of the lipid mixture, which we link to a hydrophobic mismatch between the acyl chains of the respective lipid components.





(top) Combined fit of normalized intermediate structure factor S(q,t)/S(q) with contributions from vesicle mass diffusion and bilayer undulation dynamics, respectively; (bottom) Undulation relaxation rates obtained from combined fits vs. cubed momentum transfer. Following Zilman-Granek theory, bilayer bending rigidities κ_B are derived from linear slope of these curves.

References:

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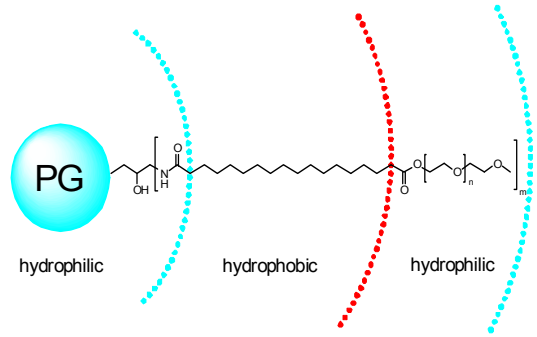
Institutions:

(1) Helmholtz Zentrum Berlin, Institut für Weiche Materie und Funktionale Materialien
(2) Universität Bayreuth, Physikalische Chemie I
(3) Institut Laue-Langevin, Time-of-Flight and High Resolution, Grenoble, France

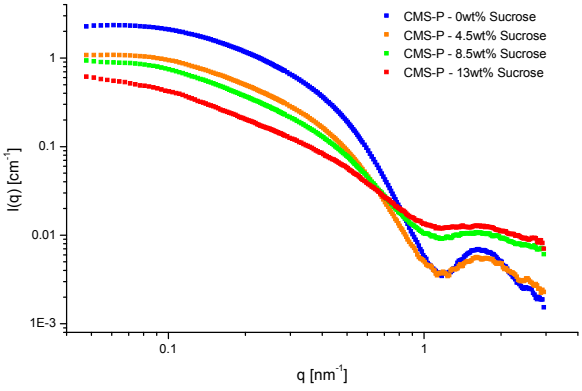
Structural Investigations aiming Core-Multishell Nanoparticles

Christian Rabe, Emanuel Fleige[1]

Liposome-like core-multishell (CMS) nanoparticles are based on polar pseudo-dendritic cores of hydrophilic poly(ethylene glycol). The core molecules are chemically linked to a surrounding double layer composed of hydrophobic alkyl chains and hydrophilic monomethyl poly(ethylene glycol) [1].
This unique structure allows the CMS molecules to be solubilized in different polar and non-polar media. Furthermore molecules of different compositions as for example anti cancer drugs can be immobilized by the CMS host structure [1, 2]. Due to their excellent encapsulation properties, non-toxicity and an ability to pass the skin barrier easily CMS-nanoparticles are seen as a promising tool for drug delivery applications [2, 3].
For an advanced understanding governing these outstanding properties a closer look to the structure and structural changes of the CMS-nanotransporters is focused on using different scattering techniques. Studies of the dynamic light scattering (DLS) underline further experiments of small angle neutron scattering (SANS) in combination with the complimentary small angle X-ray scattering (SAXS) and allows us to study these unique molecules in great detail. By simply adding sugar to an aqueous solution of CMS-molecules in the case of SAXS or by using different mixtures of D_2O and H_2O in the case of SANS experiments it is possible to tune the scattering contrast in a wide range. This technique is known as contrast variation and was successfully applied to various systems before [4, 5].
A close collaboration with our co-workers from Free University Berlin made it also possible to generate deuterated CMS-nanocarriers for contrast variation studies using SANS.



Schematic drawing of the chemical composition of a CMS-particle



Scattering curves obtained by SAXS from CMS-molecules measured at different contrasts.

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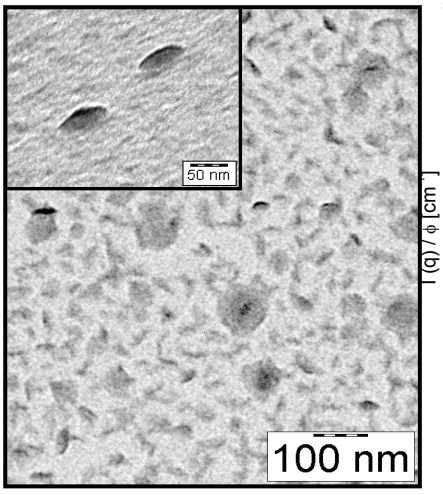
[1] Freie Universität Berlin, *Institut für Chemie und Biochemie – Organische Chemie* – AG Prof. R. Haag

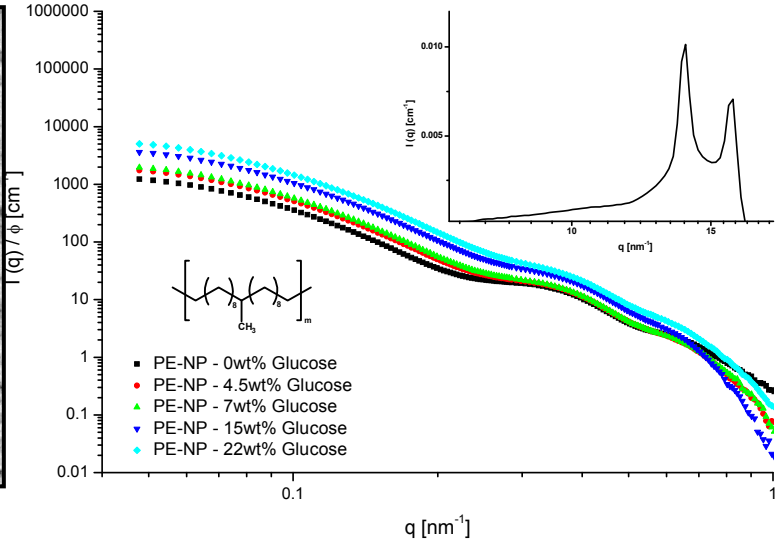
Poly(ethylene) Nanocrystals – Small Angle X-Ray Scattering Studies

Christian Rabe, Karsten Vogtt, Justyna Trzaskowski [2], Anna Osichow[2]

Poly(ethylene) (PE) is a well known and intensively studied polymer which found its way to numerous applications in daily life.
The crystallinity is one of the determinants for the resulting properties of a material. In the case of polymers the crystallinity results from van der Waals interactions between the chain segments. The formation of crystalline fractions within polymers was intensively studied over years but is not fully understood yet. The synthesis of single nanocrystals will help to overcome this lack of knowledge. Recently synthetic approaches leading to stable dispersions of single nanocrystals were successful. This includes the formation of isolated nanocrystals with well defined defect spots within the polymer crystallite. Furthermore the morphological changes of polyethylene nanocrystals during a heat treatment process are of special interest.
Structural investigations using small angle x-ray scattering (SAXS) in combination with wide angle scattering (WAXS), dynamic light scattering (DLS), cryogenic transmission electron microscopy (cryo-TEM) and calorimetric techniques (DSC) allows the determination of the nanoparticles morphology and their crystalline structure with high precision.
The contrast variation technique in addition is a suitable tool for SAXS-studies of the nanocrystals. By just adding sugar to the polymer dispersions the contrast can be varied in the way that the shape and the inner structure of the nanoparticles are accessible. A set of simulations on the so called contrast decomposition of different morphologies underline these studies.



TEM micrograph of poly(ethylene) nanocrystals containing branched PE-chains



SAXS intensities obtained from PE-nanocrystal dispersions at different contrasts. The inset shows the WAXS intensities referring to the nanoparticles crystalline phase .

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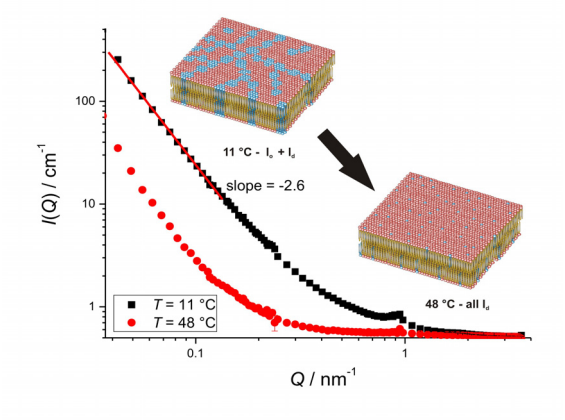
[2] Universität Konstanz, *Fachbereich Chemie* – AG Prof. S. Mecking

Small-Angle Scattering on Protein and Lipid Systems

Karsten Vogtt

Proteins and lipids are prominent examples of biological soft matter. Most proteins exhibit their stable, native form in a relatively small temperature window around room temperature. In dilute aqueous solution, they can be treated as colloidal supensions. In living beings, proteins are involved in a broad range of biological functions, serving as e.g. regulators or as catalysts. Lipids are mainly found as element of structure in the biological cell membranes. Hereby biological cell walls are not “static”, uniform entities separating the cytoplasm completely from its environment, but flexible and dynamic structures which allow local “response” to external stimuli and the controlled exchange of substances over the bilayer. It has been proposed, that local, lateral phase separation into microdomains within the lipid bilayer is involved in the occurrence of such small, functional “patches” - or “lipid rafts”, as they were termed.



Scattered intensity I(Q) of a ternary lipid mixture at two different temperatures. The strong increase of I(Q) at lower temperature is indicative for phase separation with a characteristical spatial mass distribution.

Small angle neutron scattering is an excellent tool to characterize such soft matter systems. Neutrons represent a nearly non-invasive probe, which are sensitive for the atomic nuclei rather than the electron density as it is the case for x-ray scattering. Thus the usually susceptible biological samples are not damaged. Additionally, the sensitivity for different atomic species allows the usage of the so called isotope labelling method. The in biological systems abundant element hydrogen ^{1}H can be selectively replaced by the isotope deuterium ^{2}H, which is a much stronger coherent scatterer of neutrons. Thus certain structural patterns in molecular assemblies can be selectively “labelled” and detected. This technique was used to trace phase separation into microdomains in a ternary lipid mixture according to the concept of lipid rafts, as outlined above. The aliphatic side chains of one lipid species were deuterated and small angle neutron scattering was employed to probe for inhomogenous lateral distribution of lipids within the bilayer as function of temperature (Figure). The results show that at least in such lipid model systems, small microdomains exist on length scales of nanometers and thus would exhibit the appropriate size to act as “functional patches” in biological systems.

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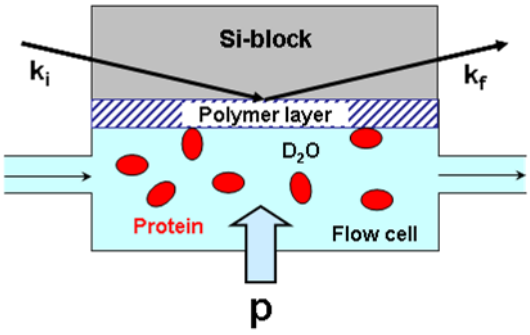
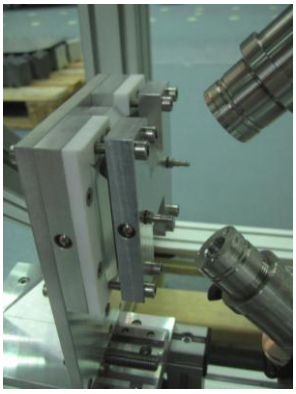
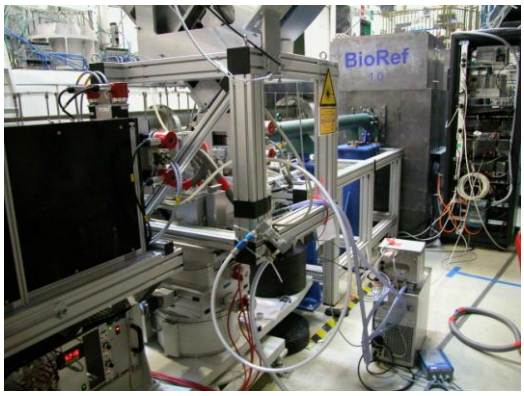
Interfaces: Beamlines and Research

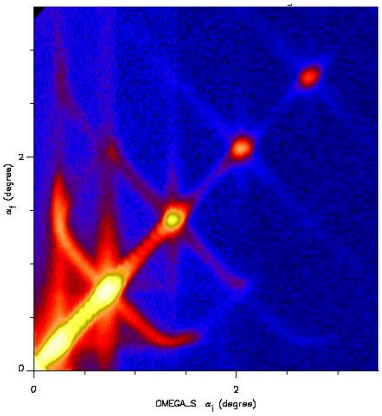
Roland Steitz



The interfaces group takes its name as its mission: The group provides active transport of knowledge between in the interior, the institute of soft matter and functional materials, and the exterior, its short and long term guests and cooperation partners from academia and industry, like a functional unit of a cell membrane. The interfaces team is responsible for the neutron reflectometer V6 and the new TOF neutron reflectometer BioRef (V18), the latter in cooperation with the Ruprecht-Karls-Universität Heidelberg (Prof. (apl.) R. Dahint, Prof. M. Grunze). The group keeps a strong interactive research profile with its short and long term cooperation partners. Within the Biophysics user laboratory (BioLab) *Interfaces* provides specialized on-site preparation techniques (Layer-by-layer deposition, spin coating, Langmuir-Blodgett and Langmuir-Schäfer deposition) and further off neutron-beamline characterization like ATR-FTIR. In addition we supply our users with complementary x-ray reflectivity and diffraction techniques.
Our research activities are committed to studies on structure and functionality of complex interfaces. At present research topics focus on bio-lubrication, responsive solid-liquid interfaces and bio-mimetic systems and their interactions with cellular components as well as on the development of dedicated instrumentation for the purpose.
In 2010 the group served 7 short term external user groups, 5 experimental campaigns from long term cooperation partners, and run 4 in-house sessions on the multipurpose neutron reflectometer V6. Together with our partners R. Dahint and M. Grunze, Ruprecht-Karls-Universität Heidelberg, we are proud to announce finalizing commissioning of BioRef and its first user experiment with C. Garvey from ANSTO, Australia, in August. As a joint activity with RKU the group established routine operation of a dedicated sample cell for neutron reflectivity investigations at solid-liquid interfaces up to 1000 bar hydrostatic pressure. Currently we are working together with partner from the University of Technology Dortmund (TUD), C. Czeslik, in expanding the accessible pressure for neutron reflectivity investigations to even 2500 bar. Together with our cooperation partner on the hard matter side, K. Temst (Catholic University Leuven, Belgium), D. Wallacher from sample environment and colleagues from the institute for complex magnetic materials at HZB, we developed and successfully used a sample cell for simultaneous polarized neutron reflectometry and anisotropic magnetoresistance measurements. A long-lasting and very successful agreement on cooperation on investigations of soft matter interfaces with the Max-Planck-Institute of Colloid- and Interface Science, Golm, ended this year. We are proud to announce that this cooperation agreement was taken up by the Institute of Physical Chemistry, University of Technology Berlin (TUB) with Regine von Klitzing.







Coworkers

Dr. Roland Steitz
Dr. Marcus Trapp
Dr. Ralf Köhler
Dipl. Phys. Martin Kreuzer
Dipl. Phys. Matthias Reinhardt
Holger Herrlich (stud.)

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1. Kreuzer, M.; Kaltofen, T.; Steitz, R.; Zehnder, B. H.; Dahint, R., Pressure cell for investigations of solid--liquid interfaces by neutron reflectivity. *Review of Scientific Instruments* **2011,** 82, (2), 023902-7.
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5. Strobl, M.; Steitz, R.; Kreuzer, M.; Nawara, A.; Paul, A.; Mezei, F.; Rose, M.; Grunze, M.; Dahint, R., BioRef – a time-of-flight neutron reflectometer combined with in-situ infrared spectroscopy at the Helmholtz Centre Berlin. *Journal of Physics: Conference Series* **2010,** 251, (1), 012059.
6. Evers, F.; Steitz, R.; Tolan, M.; Czeslik, C., Analysis of Hofmeister Effects on the Density Profile of Protein Adsorbates: A Neutron Reflectivity Study. *Journal of Physical Chemistry B* **2009,** 113, (25), 8462-8465.
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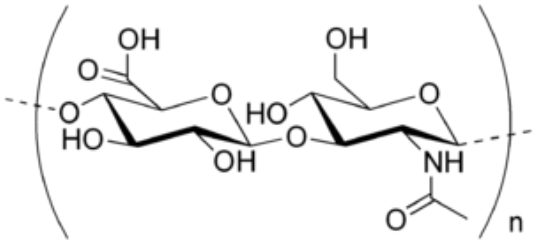
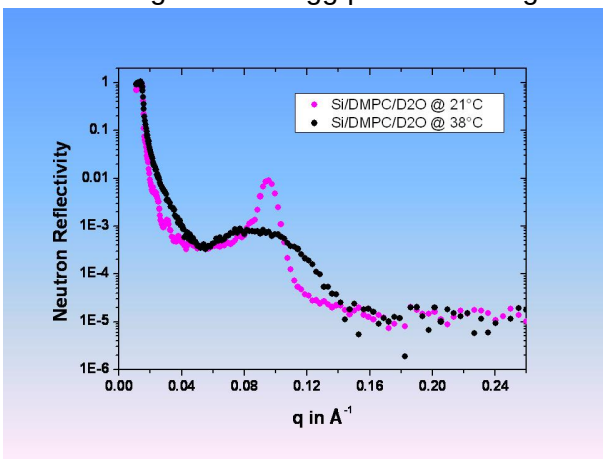
The Swelling/Stability Effect of Hyaluron on a Lipid Multilayer System

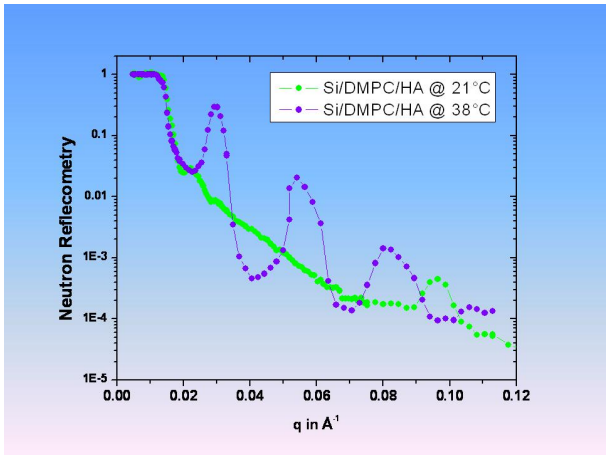
Martin Kreuzer*, M. Reinhardt, M. Strobl, R. Dahint*, R.Steitz



Hyaluron (HA) is a high molecular weight polysaccharide. HA is involved in a wide range of processes in the human body, such as wound healing [1], severe stress, tumor progression and invasion [2]. HA is also known as a lubricant in human joints [3]. Recently we were able to show, that HA also stabilizes lipid multilayer systems at physiological conditions:

Neutron reflectometry measurements in a flow cell with excess D_2O verified, that a oligolamellar DMPC lipid bilayers coating (bulk phase transition temperature T_{pt}=23°C) remained stable on a silicon substrate at 21°C in its ordered state (L_β) with a d-spacing of 66Å, but detached almost completely at 38°C in its chain-disordered L_α state from the solid support, in the figure on the right indicated by the vanishing of the Bragg peak. The origin of the loss of the oligolamellar DMPC bilayer stack at 38°C is unclear, but most likely related to the unbinding transition in the chain-disordered state of the lipid lamellae [4]. By contrast oligolamellar lipid bilayers remained stable on a substrate at 38°C when incubated with a solution of D_2O with HA: In an independent experiment, carried out at the V6 neutron reflectometer, an oligolamellar lipid bilayers stack was measured against a solution of 3mg/mL HA in D_2O. The sample was investigated shortly after incubating at 21°C and after raising sample temperature to 38°C. The oligolamellar lipid layer remained stable on the substrate, but an immense swelling occurred until a d-spacing of 209Å, as indicated in the bottom right figure.





In the literature there is no consensus about how a polysaccharide, e.g. HA, affects the lipid phase behavior. The two differing hypotheses are: that (i) the swelling is caused by direct interactions between the solutes and the lipids; or that (ii) the swelling is originated by nonspecific effects related to the osmotic properties of the solutes. Further work is in progress for deriving a clear discrimination and final answer to that open problem.

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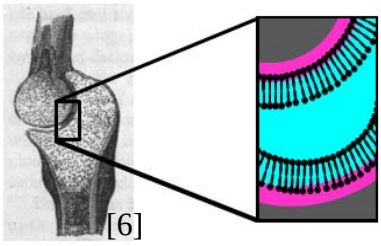
* Ruprecht-Karls-Universität Heidelberg

Lubrication in natural joints – a shear dependence study

M. Kreuzer*, M. Strobl, M. Reinhardt, R. Dahint*, R. Steitz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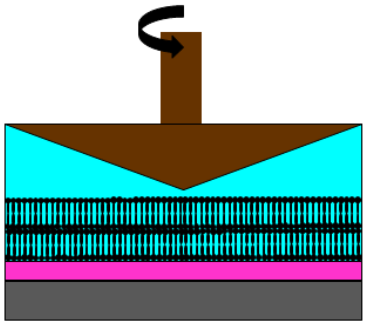

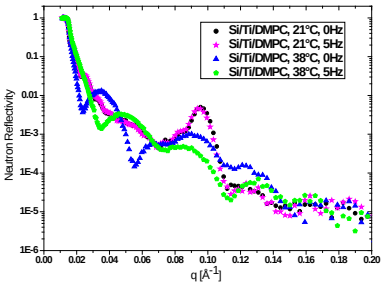

The search for biocompatible materials has become one of the key issues in modern medicine. Coating of implants by lipid layers is widely used for a better acceptance of the implant in the human organism. With no coverage of their surface artificial implants would invoke macrophage reactions immediately. While on the one hand the durability of implants has been improved significantly, on the other hand the need for permanent and more long-lasting implants is steadily growing [1]. For biomedical applications, titanium-based alloys such as Ti-6Al-4V are most suitable [2]. In case of movable and mechanically stressed implants, such as artificial joints, lubrication under pressure and shear has to be optimized in addition to biocompatibility aspects. While in early artificial joints the movable parts directly contacted each other, researchers nowadays try to copy the principles of lubrication observed in natural joints to reduce friction [3]. Here, the two surfaces of the joint are separated by a liquid phase, the synovial fluid, which mainly contains hyaluronic acid (HA). The most relevant mechanisms and physicochemical parameters to reduce friction are still unclear and subject of controversial discussions. Many studies emphasize the importance of HA for joint lubrication. Furthermore surface-active lipids, which cover the contact areas of natural joints, are considered to play an important role in the reduction of friction [4].

We represented such interface by a suitable model system and employed neutron reflectometry (NR) to study its structural features using a shear setup. The model system was designed as a soft supported lipid membrane (80% POPC + 20% POPS), one bilayer, on the top of a water-swollen polyelectrolyte multilayer on silicon support and incubated in a 3mg/mL solution of HA with D_2O. Our measurements revealed, that an HA-layer of 38Å thickness adsorbs on top of the lipid bilayer. When a shear rate of 2Hz was applied, the HA-layer decreased in thickness to 30Å. Also the lipid membrane thickness decreased from 41Å to 33Å. The same system measured against a pure D_2O did not change its characteristics. Thus, our experiments show that only the combined system of a lipid layer in contact with HA-solution changes, when a shear force is applied.

NR measurements were continued on oligolamellar lipid bilayers directly prepared on a titanium coated silicon substrate, the latter serving as a model for a metallic implant. As seen from the corresponding reflectivity curves, the oligolamellar system became unstable with higher temperature and shear. Further work is needed to clarify the importance of combining of metallic implant surfaces with lipid coatings for forthcoming implant modifications [5].

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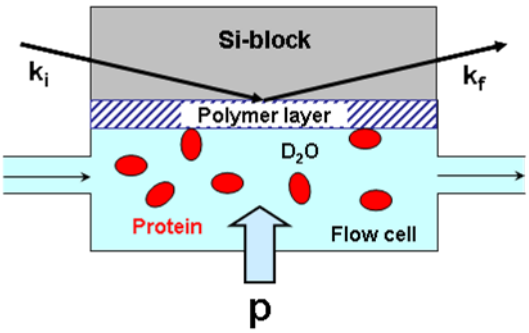
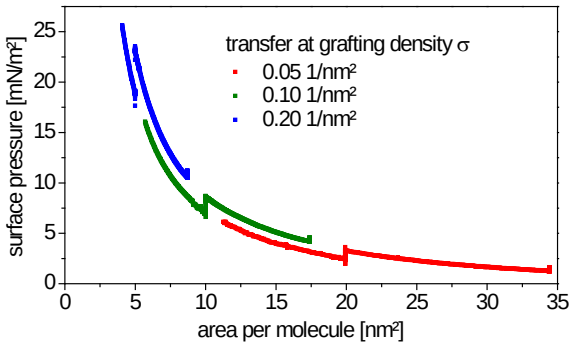
* Ruprecht-Karls-Universität Heidelberg

Functional Interfaces – Brushes and Pressure

Matthias Reinhardt, Martin Kreuzer*, Roland Steitz



High pressure is an important feature of certain natural membrane environments and proteins, for instance in the context of marine biotopes. In that case pressure induced unfolding and denaturation of proteins is of outmost importance. As do natural lipid membranes also polymer brushes provide a soft interface for adsorbed proteins without changing their functionality. We studied adsorbed proteins (BSA) on polymer brushes (dPS-PAA) of different grafting densities at the solid-liquid interface at elevated hydrostatic pressure. Due to the high transparency of solid crystalline materials for neutrons and its high spatial resolution neutron reflectivity (NR) is a perfect tool for investigating structural changes at these solid-liquid interfaces on the nanometer scale when high pressure is applied.

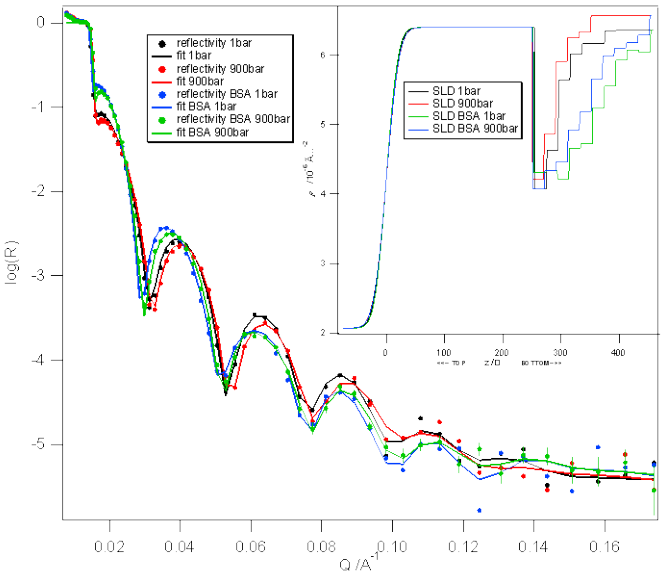


We succeeded in transferring precursor Langmuir films of deuterated poly(styrene)-b-poly(acrylic acid) block copolymers (dPS-PAA) on deuterated poly(styrene) (dPS) pre-coated silicon substrates via Langmuir-Schaefer technique. For this purpose the surface pressure of the precursor Langmuir film provides control of the grafting density and the transfer ratio of the brush as shown in the graph to the left.

The NR measurements were conducted on V6 at HZB and AMOR at SINQ/PSI.

We found small reversible changes in the reflectivity at elevated pressure. These changes are mainly contributed to an increased SLD of the D_2O subphase. We also found an increased amount of adsorbed BSA proteins for higher grafting densities of the brush.

Elevated hydrostatic pressure up to 900bar does not show significant measureable changes of the adsorbed proteins.



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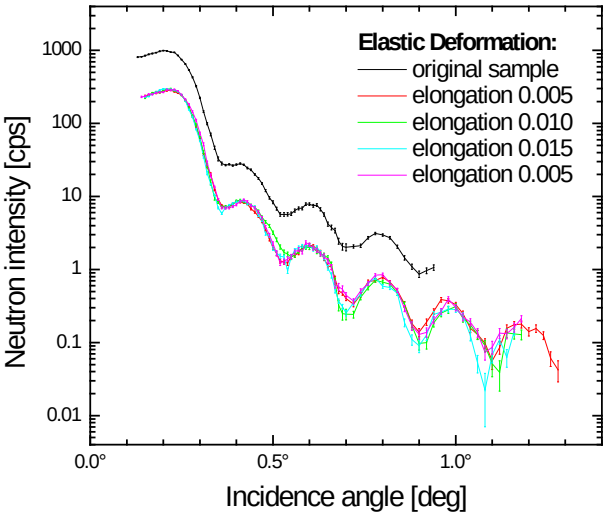
* Ruprecht-Karls-Universität Heidelberg

Effect of Uniaxial Strain of Polyelectrolyte Multilayers

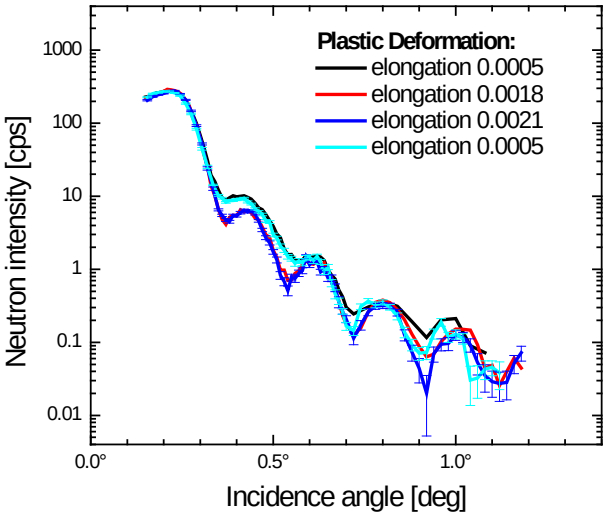
Johannes Früh*, Helmuth Möhwald*, Ralf Köhler+

Since there introduction by Decher et al. [1] Polyelectrolyte Multilayers (PEM) have attracted a great scientific interest. The reasons for that are numerous. PEM are easy to prepare on water-born chemistry, the preparation allows for adjusting of thickness and roughness on nanometer scale and with a high reproducibility. PEM are sustainable, a functionalization is possible, and e.g. PEM allow for incorporation of materials (e.g. particles, or even living cells have been incorporated into a PEM matrix) [2].



A key feature for application of PEM is their mechanical behaviour (e.g. stress-strain, or fatigue). Additionally, tests of the mechanics can give information on structure and structural changes of PEM which are not fully understood yet. After having investigated the change of the internal structure for large deformations up to 10% [3], now, we focus on studying effects for small elongations. The aim is to learn about the crossover of reversible and irreversible processes inside the PEM during deformation.

Our new approach is, instead of loading rubber-supported PEM film [3,4,5], to bend a PEM coated thin solid glass substrate. This way a one-dimensional stress is applied [6]. The bent samples are investigated by specular neutron reflectometry whereby the irradiation is perpendicular to the bending axis. Only the specular reflected beam on top of the bent sample is analyzed.



First results were archieved with a PSS/PDADMAC polyelectrolyte system (polystyrene sulfonate / polydiallyldimethyl ammonium chloride) prepared by spraying technique. We found the transition from elastic to plastic behaviour at a very low elongation of ca 0.2%. This is a value typical for solids like aluminium or copper. For polymers on would expect a higher value. Additionally this plastic deformation comes with a slide increase of the film thickness. Both findings are very surprising and seem, at first glance, to conflict each other. Future studies are planed to clarify these points.

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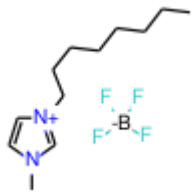
* MPI-KG, Potsdam/Golm, +TU Berlin

Anisotropic Fluids at Solid Interfaces: Ionic Liquids

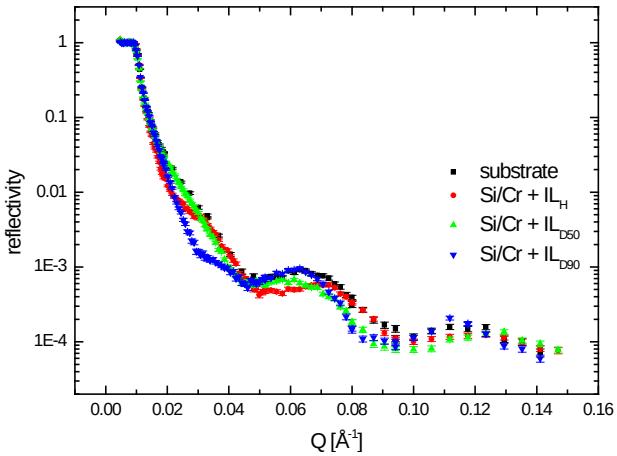
Ralf Köhler*, Rumen Krastev+, Benilde Saramago#

Ionic interactions are the strongest interactions on molecular level, thus materials which are mainly governed by ionic bonds are typically solid a room temperature. Inorganic salts usually also possess ordered crystal lattices. But Ionic Liquids (IL) behave different. They usually have a strong asymmetry in ion size and exhibit, for one ion at least, a relative complex chemical composition. This asymmetry in physico-chemical built-up comes with a broad variety of unusual (macroscopic) properties. The most obvious property was eponymous: Ionic Liquids are liquid salts at room temperature [1,2]. The complex interactions in the ILs yield to complex behaviour, which make IL applicable for many interesting tasks: as lubricants, catalysts, electrolytes (batteries), solvents and dispersants [3,4]. Beside that IL are interesting matters for fundamental science.



For most of the applications, mentioned above, the interfacial interactions play an important role. This is the motivation to study the wetting behaviour of IL at solid, in this case metallic interfaces, whereby a possible ordering was of special interest. We addressed this topic by using three species of 1-methyl-3-octylimidazolium tetrafluoroborate [OMIM][BF4] which have a different degree of deuterium (non, 50% and 90%) in the ring of the cationic OMIM-molecule. This labelling would allow for determination of a possible layering of the IL along the OMIM-axis and perpendi-cular to the surface. Only a few scattering studies address this topic [5,6].

We found evidence for a layered structure in vicinity of the solid surface. The reflectometry curves show different shape although the thickness of the liquid film is almost the same. This study was paralleled by AFM measurements which confirmed the existence of layers parallel to the surface, but, at the same time, gave hints for a more complex structuring at the surface. Most likely ordered and unordered OMIM-layers coexist at different distance towards the solid interface. Further investigations are necessary to clarify these findings and to allow for establishing a final model of the structural behaviour close to the solid interface.



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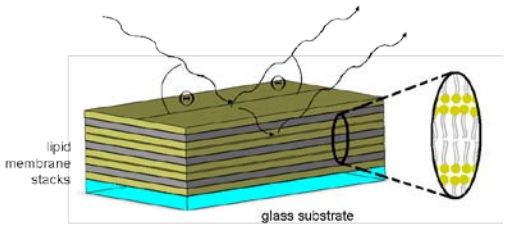
*MPI-KG Potsdam/Golm, +NMI an Universität Tübingen, Reutlingen, #TU Lisboa, Lissabon

Influence of Trehalose on the Nano-Structure of Lipid Membranes

M. Gast, U. Fattler*, T. Haußß, H. Haas*, R. Steitz

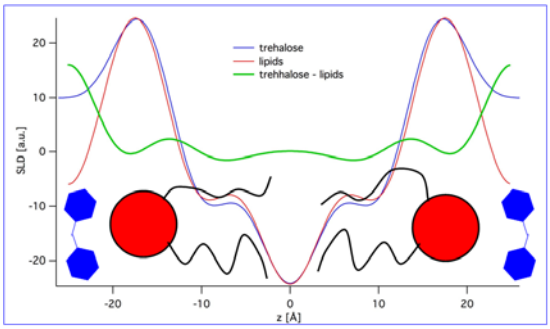


The disaccharide trehalose is known to stabilize cells and their components during drying and freezing processes and it is an important cryo- and lyoprotective excipient in biopharmaceutical manufacturing. For the very reason we investigated the influence of trehalose on the structure of lipid model membranes by x-ray- (XRD) and neutron diffraction (ND) measurements during which we simulated a drying and rehydration process of the lipid membranes. Our aim was to provide a basis for better understanding and controlling liposome dehydration processes like freeze drying or spray drying.

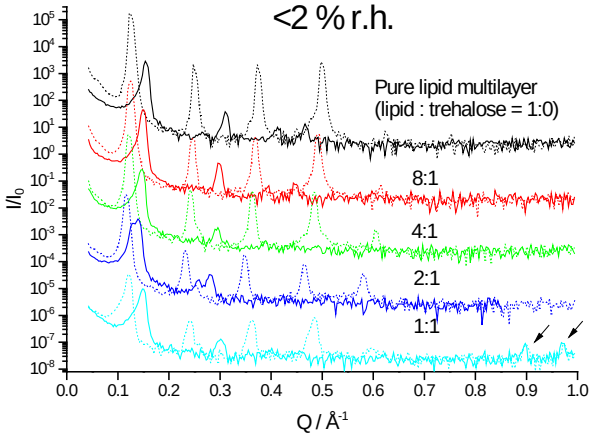
Multilamellar vesicles (MLVs) consisting of 1,2-dioleyl-sn-glycero-3-trimethyl-ammonium-propane (DOTAP) and 1,2-dioleyl-phosphatidylcholine (DOPC) were prepared with solutions of different trehalose concentrations, deposited on solid substrates and dried. The obtained stacks were then investigated by XRD and ND under controlled relative humidity (r. h.).

In the presence of trehalose, the lamellar spacing was larger than for pure lipid membranes, indicating that the trehalose inserted in headgroup region of the lipid bilayers.

By neutron scattering measurements with D_2O/H_2O contrast variation we determined the scattering length density profile of the lipid bilayers across their unit cell. From those measurements the localization of water and trehalose molecules was quantitatively deduced: The figure on the right shows that trehalose induced an increase of the SLD in the hydrophilic slab of the bilayers, but it did not penetrate into the lipid bilayer tails region (green line). We found that 2-3 water molecules per lipid headgroup are displaced by trehalose.



X-ray reflectivity measurements /s. below) permitted the illumination of structural implications of dehydrating - rehydrating DOTAP/DOPC membranes in the presence of trehalose. Up to a molar fraction lipid:sugar of 4:1, trehalose was found to insert into DOTAP/DOPC membranes as an integral part of the multilayer stack, which was not excluded from the membrane interface on repeated de- and rehydration. The resulting increase of minimum d-spacing can be considered favourable for preventing liposome fusion or aggregation. Full reversibility of structural reorganizations as a function of relative humidity thus nominates DOTAP/DOPC liposomes in trehalose robust regarding dehydration protocols in the course of manufacturing and application of pharmaceutical DOTAP/DOPC liposome products.



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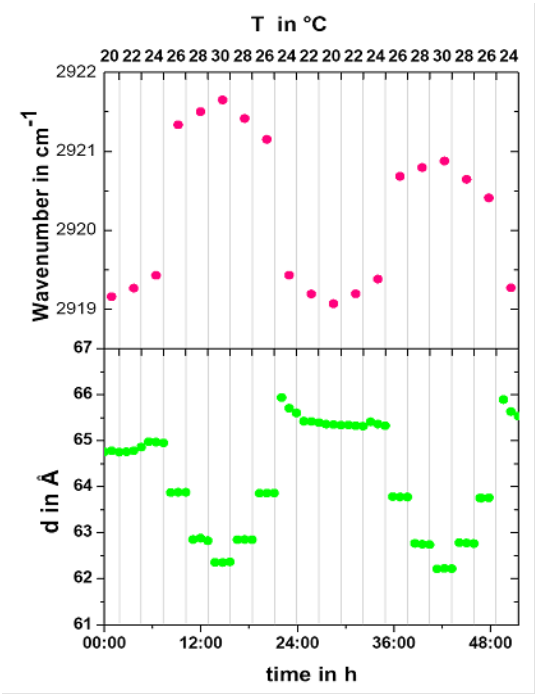
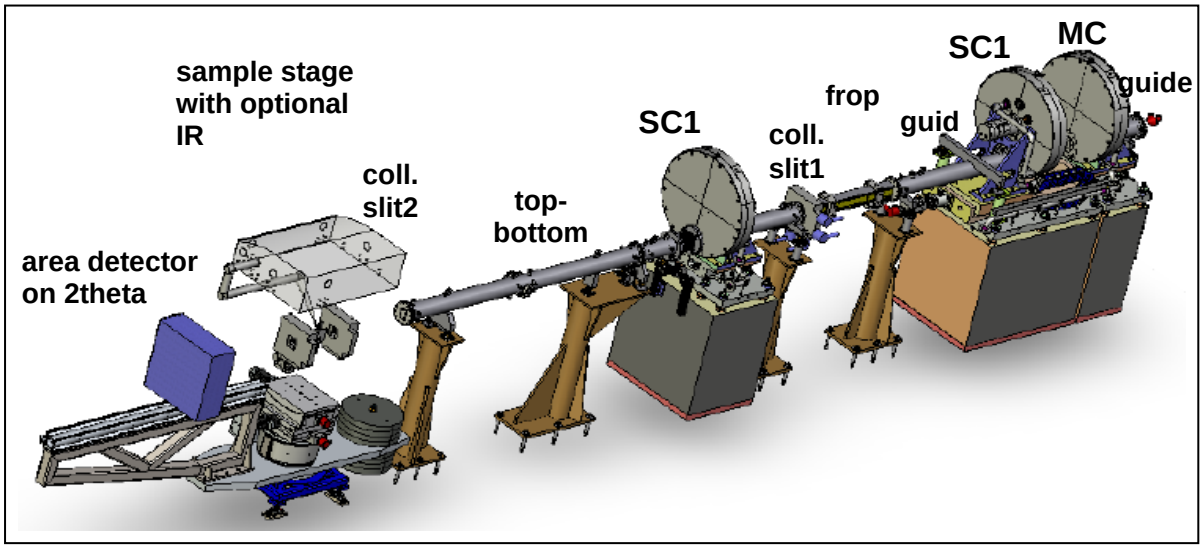
* Medigene AG, Martinsried

V18 BioRef – a Versatile Tool for Surface/Interface Characterizations

Markus Strobl, Martin Keuzer*, Reiner Dahint*, Roland Steitz

BioRef is a time-of-flight neutron reflectometer, has recently been realized in the framework of a BMBF funded project in cooperation with the University of Heidelberg. The instrument, which became operational early 2010 is mainly dedicated to investigations of solid-liquid soft matter surfaces and interfaces, which serve as model systems of Biological systems. For this purpose sample environment capable to mimic physiological conditions in terms of temperature, flow and shear as well as pressure has been and is further developed. Besides the flexibility of the instrument, which allows for tailoring instrumental conditions, like resolution and utilized wavelength band to the specific requirements of investigations and hence to enable even kinetic studies in selected scattering vector ranges, the set-up offers the unique option of in-situ attenuated total reflection Fourier transformed infrared (ATR-FTIR) spectroscopy. Such spectroscopy can be utilized to gain conformational information complementary to the structural data concerning the scattering length density (SLD) profiles deduced from the neutron reflectivity. This way e.g. the unfolding of proteins which indicates a loss of their functionality at investigated surfaces can be observed under the very same conditions under which the structural data is collected even when such conditions are not constant with time, i.e. under kinetic conditions. A first combined neutron-IR study of a lipid multilayer during a temperature scan is presented in the Figure on top. The Figure on the bottom of the page is a drawing of the principal instrument layout featuring the IR spectrometer on top of the sample position.





[1] M. Strobl, R. Steitz, M. Kreuzer, A. Nawara, A. Paul, F. Mezei, M. Rose, M. Grunze and R. Dahint, *Journal of Physics: Conference Series* **2010**, *251*, 012059.
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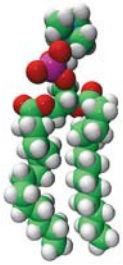
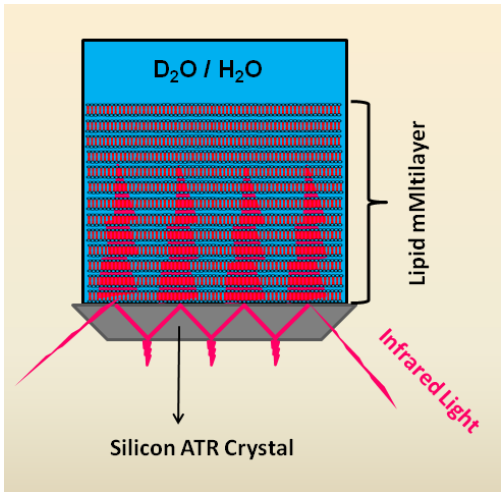
* Ruprecht-Karls-Universität Heidelberg

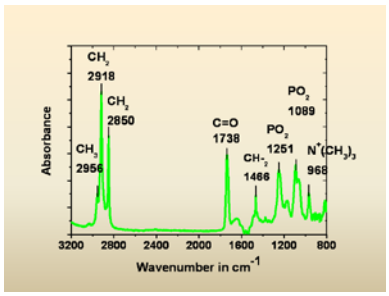
Fourier Transform Infrared pectroscopy (FTIR) at Solid-Liquid Interfaces

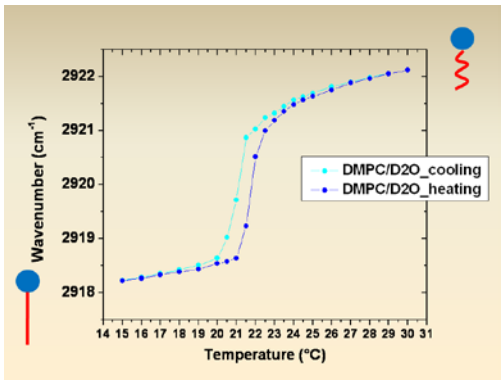
Martin Kreuzer*, Marie Charlotte Hemmer, Reiner Dahint*, Roland Steitz



Due to their hydrophilic head and hydrophobic tail groups lipid molecules are able to form multilayer membranes in an aqueous environment. Up to 40% of the molecules in cell membranes are lipids [1]. These biological membranes are essential for directed and proper molecular life functions [2]. Fundamental for understanding the functional properties of membrane lipids is a detailed knowledge of their preferred molecular conformations. Attenuated total reflectance (ATR) - FTIR is the favored technique for examining directly multilayer membranes in an aqueous environment. We utilized a BioATR II setup from Bruker Optics, where an infrared beam gets reflected several times at a silicon-water interface, before it is detected with a nitrogen cooled, mercury cadmium telluride (MCT) detector. The infrared absorbance signal from a lipid multilayer, attached to the silicon surface and incubated with an aqueous solution, is enhanced with every reflection at the interface. In addition, the temperature controlled setup made it possible to measure the confirmation of lipid membranes in a wide range of temperatures against different aqueous solutions. In particular the CH_2 vibrations of the tail groups of the lipids gave information about their lamellar phase (e.g. gel-like $P_{\beta'}$ or fluid-like L_α phase) [3].







The temperature dependent measurements in the ATR-FTIR setup with a lipid multilayer against excess D_2O showed a shift of the symmetric and anti-symmetric CH_2 stretching band at the phase transition. For the lipid molecule 1,2-Dimyristoyl-sn-Glycero-3-phosphocholine (DMPC) the symmetric CH_2 stretching band shifted from 2850cm^{-1} to 2853cm^{-1}, corresponding to the $P_{\beta'}$ and L_α phase, respectively, when the temperature was scanned between 15°C and 30°C. The asymmetric CH_2 stretching band shifted from 2918cm^{-1} to 2923cm^{-1}. The most pronounced shift occurred at 21.5°C, which is close to the phase transition of the bulk lamellar phase of DMPC in H_2O at app. 24°C.

The design of an advanced ATR-FTIR setup including a sample cell made it possible to perform a combined measurement of Neutron Reflectivity and ATR-FTIR on the same sample, at the same time. This unique setup is realized at the BioRef neutron beamline at the HZB. Here, conformational as well as structural information of the interface can be measured.

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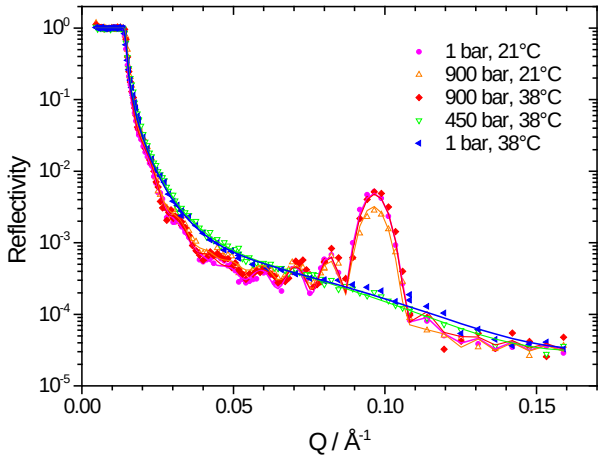
Pressure cell for investigations of solid-liquid interfaces by neutron reflectivity

Martin Kreuzer*, Thomas Kaltofen*, Beat H. Zehnder#, Reiner Dahint*, Roland Steitz

Studies of the high pressure phase behaviour of lipid and surfactant systems, in particular of phospholipid bilayers, which can serve as model biomembranes, have become a standard setup in bulk systems.[1] Sample cells for hydrostatic pressure conditions up to 7000 bar for use with neutron and X-ray diffraction techniques are available nowadays.[2], [3] Design and manufacturing of those cells is favoured by the small sample volume, typically 0.04-0.5 mL, needed for the diffraction experiments. Cross sections of these pressure cells are of the order of a few mm^2 and thus easy to handle up to highest pressures. Reflectivity studies on confined systems and even single lipid membranes immobilised at solid-liquid interfaces on the opposite require large surface areas typically of the order of some tens of cm^2 and some tens of mL liquid volume. These pre-conditons hindered complementary investigations of surface bound systems for very long.

We recently succeeded in developing an apparatus for measuring scattering length density and structure of molecular layers at planar solid–liquid interfaces under high hydrostatic pressure conditions (figures).[4] The device is designed for in situ characterizations utilizing neutron reflectometry in the pressure range 1–1000 bar at temperatures between 5 and 60 °C. The pressure cell is constructed such that stratified molecular layers on crystalline substrates of silicon, quartz, or sapphire with a surface area of 2800 mm^2 can be investigated against noncorrosive liquid phases. The large substrate surface area enables reflectivity to be measured down to 10^{-5} (without background correction) and thus facilitates determination of the scattering length density profile across the interface as a function of applied load. Our current interest is on the stability of oligolamellar lipid coatings on silicon surfaces against aqueous phases as a function of applied hydrostatic pressure and temperature but the device can also be employed to probe the structure of any other solid–liquid interface.



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* Ruprecht-Karls-Universität Heidelberg
# SITEC-Sieber *Engineering* AG, Ebmatingen

Colloid Chemistry

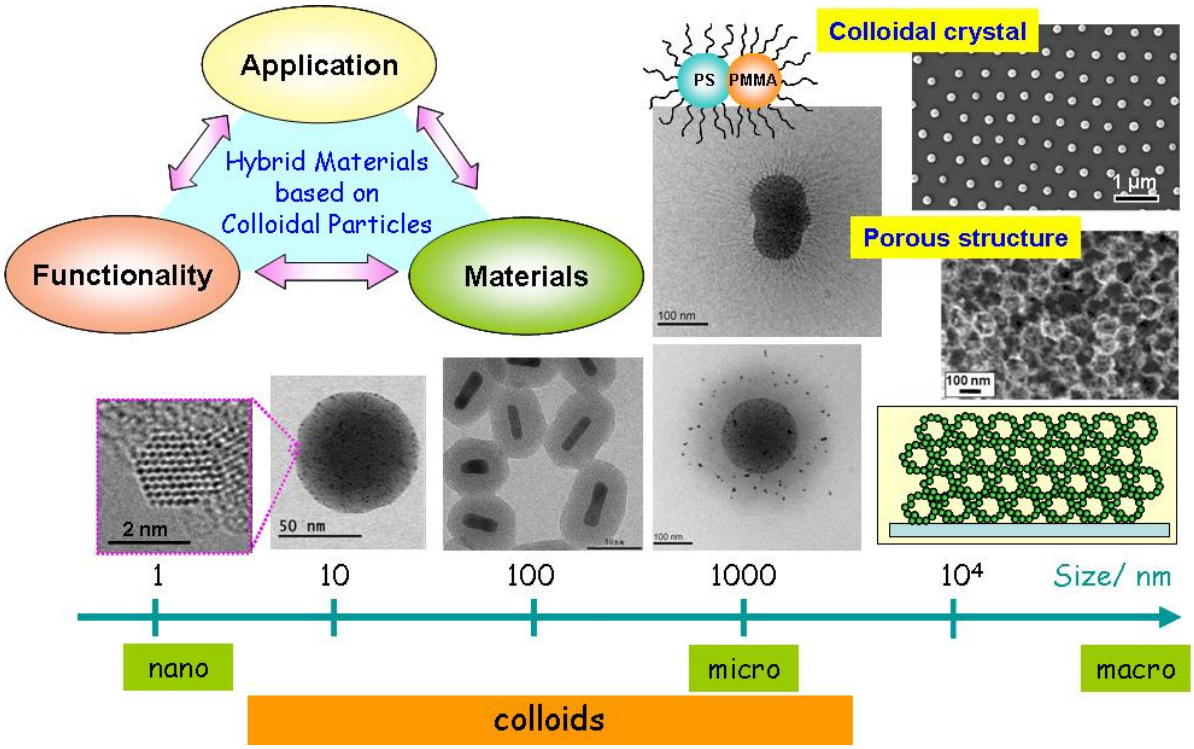
Yan Lu

The research work in the Colloid Chemistry Group mainly focuses on the design and fabrication of functional hybrid materials based on colloidal particles with versatile applications, such as catalysts, solar cells, and optical devices. Moreover, this group will run the new cryo-TEM located at the Joint Laboratory of Structural Research and provide the necessary service to the users of this instrument.

Composite particles based on metal nanoparticles (Au, Ag, Pd, Pt, etc.), metal nanoalloys (Au-Pt, Au-Pd, etc.) and metal oxide (TiO_2, ZrO_2, MnO_x, etc.) particles using colloidal particles as carrier system have been prepared. Compared to other reported carrier systems, colloidal particles have various merits, such as superior stability, facile synthesis for industrial potential, good control over particle size and composition, and easy functionalization providing novel properties. Thus, these composite particles have multiple functionalities with improved physical and chemical properties in a feasible way. These nanocomposite particles have been proven as excellent (photo)catalysts for various chemical catalytic reactions that proceed in aqueous solutions or in two-phase systems. Kinetic studies of catalytic reactions are an essential part of our research in order to understand the mechanism of the reaction in the presence of metal nanoparticles.

Special efforts have been made for the preparation of anisotropic particles, which show interesting properties in light scattering and plasmon absorption. For example, dumbbell-shaped colloids with a size around 200 nm can be prepared by seeded-emulsion polymerization, which can be used as core for the further deposition of well defined water soluble polyelectrolyte brushes or stimuli-responsive shell. On the other hand, hybrid structures based on Au nanorods can be applied as model system to study the plasmon effect of metal nanoparticles on the kinetics and efficiency of photoelectrocatalysis. In addition, possible applications like a surface plasmon polariton laser will be investigated.

Cooperation with research groups in the field of solar cells, plasmonics and catalysis within and outside HZB will help us not only to expand the application spectrum of composite particles but also result in vital understanding of the fundamentals.



Coworkers:
Dr. Yan Lu
Dipl.Chem. Julian Kaiser
Dipl.Chem. Stefanie Wunder
M.Sc. Fangfang Chu
Dipl.Chem. Andreas Ott

Selected Publications in 2009-2012:

1. S. Wu, J. Dzubiella, J. Kaiser, M. Drechsler, X. Guo, M. Ballauff, Y. Lu, "Thermosensitive Au-PNIPA Yolk-Shell Nanoparticles with Tunable Selectivity for Catalysis", *Angew. Chem.* **2012**, *DOI: 10.1002/anie.201106515*.
2. F. Polzer, S. Wunder, Y. Lu, M. Ballauff, "Oxidation of an Organic Dye Catalyzed by MnOx Nanoparticles", *J. Catal.* **2012**, *DOI: 10.1016/j.jcat.2012.01.016*.
3. Y. Lu, M. Ballauff, "Thermosensitive Core-Shell Microgels: From Colloidal Model Systems to Nanoreactors", *Prog. Polym. Sci.* **2011**, 36, 767-792.
4. S. Wunder, Y. Lu, M. Albrecht, M. Ballauff, "Catalytic activity of facetted gold nanoparticles studied by a model reaction: Evidence for substrate-induced surface restructuring", *ACS Catalysis* **2011**, 1, 908-916.
5. F. Polzer, J. Heigl, C. Schneider, M. Ballauff, O. Borisov, "Synthesis and Analysis of Zwitterionic Spherical Polyelectrolyte Brushes in Aqueous Solution", *Macromolecules* **2011**, 44 (6), 1654-1660.
6. Y. Lu, J. Yuan, F. Polzer, M. Drechsler, J. Preussner, "In-situ Growth of Catalytic Active Au-Pt Bimetallic Nanorods in Thermo-Responsive Core-Shell Microgels", *ACS Nano* **2010**, 4 (12), 7078–7086.
7. Y. Lu, T. Lunkenbein, J. Preussner, S. Proch, J. Breu, R. Kempe, M. Ballauff, "Composites of Metal Nanoparticles and TiO_2 immobilized in Spherical Polyelectrolyte Brushes", *Langmuir* **2010**, 26 (6), 4176–4183.
8. S. Wunder, F. Polzer, Y. Lu, M. Yu, M. Ballauff, "Kinetic Analysis of Catalytic Reduction of 4-Nitrophenol by Metallic Nanoparticles immobilized in Spherical Polyelectrolyte Brushes", *J. Phys. Chem. C* **2010**, 114 (19), 8814-8820.
9. M. Hoffmann, M. Siebenbürger, L. Harnau, M. Hund, C. Hanske, Y. Lu, C. S. Wagner, M. Drechsler, M. Ballauff, "Thermoresponsive colloidal molecules", *Soft Matter* **2010**, 6,1125–1128.
10. F. Polzer, D. A. Kunz, J. Breu, M. Ballauff, "Formation of Ultrathin Birnessite-Type Nanoparticles Immobilized on Spherical Polyelectrolyte Brushes", *Chem. Mater.* **2010**, 22, 2916-2922.
11. Y. Lu, M. Drechsler, "Charge-induced Self-Assembly of 2-Dimensional Thermosensitive Microgel Particle Patterns", *Langmuir* **2009**, 25, 13100-13105.
12. Y. Lu, S. Proch, M. Schrinner, M. Drechsler, R. Kempe, M. Ballauff, "Thermosensitive Core-Shell Microgel as a "Nanoreactor" for Catalytic Active Metal Nanoparticles", *J. Mater. Chem.* **2009**, 19, 3955 - 3961.
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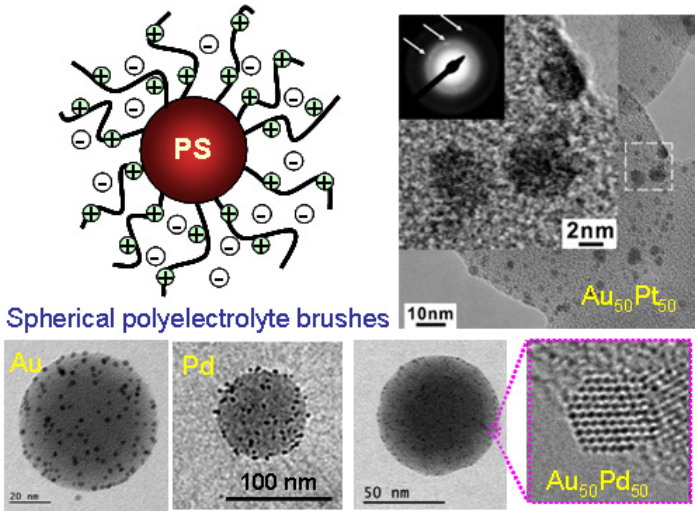
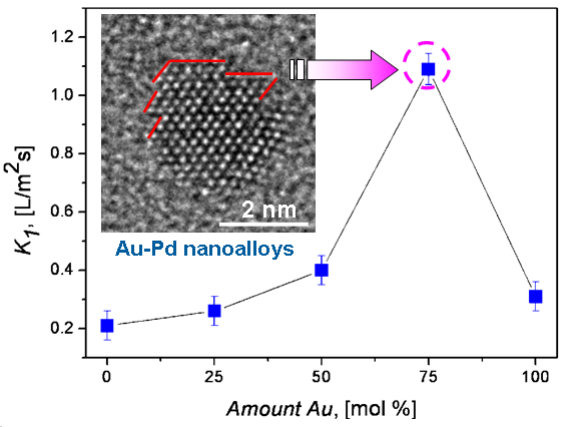
Spherical Polyelectrolyte Brushes as "Nanoreactors" for Metal Nanoparticles or Nanoalloys

Julian Kaiser, Yan Lu, M. Albrecht (Leibniz-Institut für Kristallzüchtung, Berlin), S. Kümmel (Universität Bayreuth)



Metal nanoparticles and nanoalloys have been raised a lot of interests in the present. The biggest problem is the prevention of metal nanoparticles from agglomeration. During our study, spherical polyelectrolyte brushes (SPB) have been used as "nanoreactors", in which metal nanoparticles can be immobilized and handled in an easier fashion. The SPB particles consist of a polystyrene core, onto which long linear chains of polyelectrolyte are densely grafted. During our study, various nanoparticles of noble metals (such as Au, Pd, Pt, etc.) can be immobilized in this way and used for catalysis in aqueous media, that is, under very mild conditions.

Moreover, metal nanoallys have attracted more interests than metal nanoparticles recently due to the fact that their chemical and physical properties may be tuned by varying the composition and atomic ordering. During our research, we have demonstrated that SPB can work efficiently as a carrier system for the immobilization of metal nanoalloys (such as Au-Pt, Au-Pd). In this case, with the help of high resolution transmission electron microscopy (HR-TEM), powder x-ray diffraction (XRD) and extended x-ray absorption fine structure (EXAFS), it is possible for us to get precise information about the dynamics and compositional and structural evolution of these metal nanoalloy particles. In addition, the catalytic activity of the generated nanoalloys has been tested by the reduction of 4-nitrophenol. The rate constant normalized to metal surface area goes through a maximum for the catalytic reduction as the function of Au amount in bimetallic nanoparticles, which indicates a synergistic effect of both metals related to their catalytic activity.



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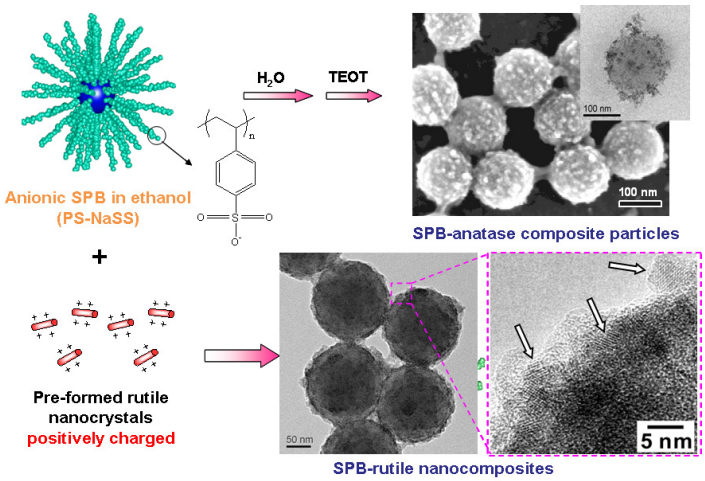
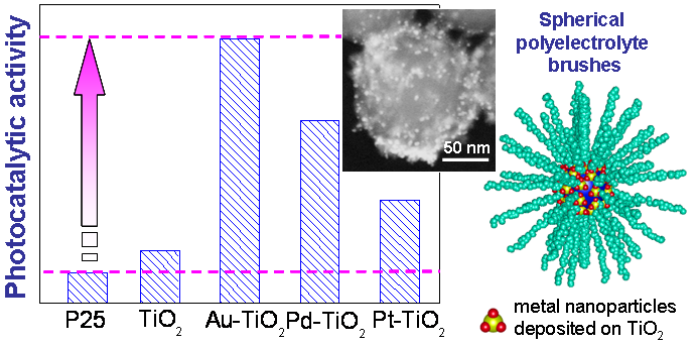
Spherical Polyelectrolyte Brushes as "nanoreactor" for Well-Defined Crystalline TiO_2 Nanoparticles

Yan Lu



TiO_2 nanomaterials have received much attention recently due to their photocatalytic activity, high chemical stability and possible applications in solar cells. Moreover, mesoporous TiO_2 networks with high surface area are of particular interest for a number of applications. Colloidal latex particles have been used for the preparation of hollow TiO_2 spheres or continuous macroporous TiO_2 structures. However, all as-prepared TiO_2 composites prepared in this way by a sol-gel approach are amorphous. The latex particles act only as a template for the macroporous structure and calcination is required to achieve sufficient crystallinity.

Spherical polyelectrolyte brushes (SPB) particles may serve as well-defined nanoreactors for the immobilization of TiO_2 nanoparticles. The SPB particles consist of a solid PS core from which long anionic polyelectrolyte chains are densely grafted. Crystalline anatase or rutile TiO_2 nanoparticles can be generated directly by "sol-gel" method or electrostatic adsorption in the presence of SPBs at low temperature, respectively. Thus, composite particles of a polymeric carrier and crystalline TiO_2 in a well-defined modification can be obtained without any further heat treatment. In addition, the as-prepared TiO_2 nanocomposites exhibit an excellent colloidal stability. The photocatalytic activity of the composite particles for the degradation of dye RhB under UV irradiation can be dramatically enhanced after deposition of metal nanoparticles on it. Finally, calcination of the composite particles leads to a macroporous scaffold of mesoporous TiO_2 nanoparticles, which are thermally stable against collapse. Possible applications, as e.g. for solar cells, have been proved.



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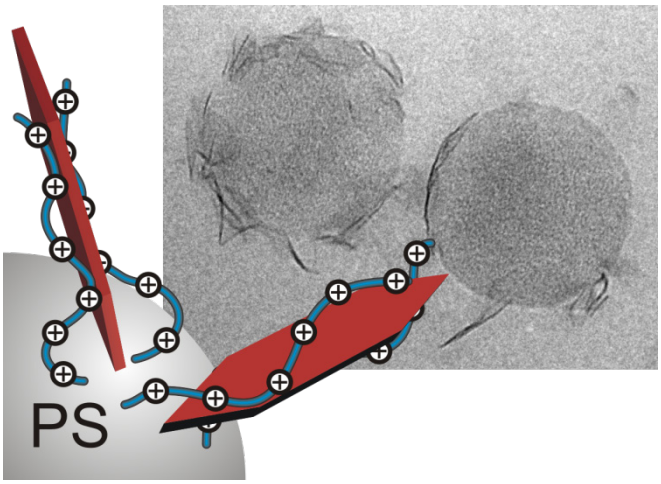
Fabrication and Catalytic application of Ultrathin Birnessite-Type Nanoparticles Immobilized on Cationic Spherical Polyelectrolyte Brushes

Frank Polzer, Stefanie Wunder

Layered manganese oxide materials attracted much interest because of their potential applications as catalysts and electrode materials. In both fields, high surface to volume ratios are favored. For layered materials, multistep processes like intercalation of bulky ions with subsequent delamination are needed to obtain thin- or monolayered structures with high surface areas.



We present a new method of in situ formation and stabilization of ultrathin, layered manganese oxide nanoparticles (MnO_xNP) in aqueous solution by using spherical polyelectrolyte brush particles (SPB) without any further reducing agent and delamination procedure.

A combination of TEM, cryoTEM, PXRD, EDX and XAS studies was used for detailed characterization. The nanoparticles are of birnessite type, a layered material composed of hydrous lamellae of hexagonal MnO_x sheets. These sheets form nanoparticles with an average length of 20 nm and a breadth of ca. 1.6 nm indicating a composition of single lamellae or of ultrathin stacks of very few lamellae. The individual layers have a stacking disorder leading to hk bands in the PXRD pattern which is typical for c*-disordered K^+ birnessite. XAS proved to be an excellent way to gain important information on the poorly crystalline nanoparticles such as the interatomic distances, the coordination chemistry and the average oxidation state. Furthermore, the nanoparticles are well stabilized against coagulation by immobilization onto the SPB carrier particles in aqueous solution. The catalytic activity of these composite particles was investigated using the oxidation of morin by hydrogen peroxide as a model reaction. The oxidative degradation of morin was followed by UV/vis spectroscopy leading to an apparent rate constant k_{app}. We propose a modeling of the results in terms of a Langmuir-Hinshelwood model. k_{app} can be related to the kinetic constant k and to the apparent adsorption constants of H_2O_2 and morin. Based on this model, the dependence of k_{app} on temperature can be traced back to the activation energy of the rate constant k and the adsorption enthalpies of both educts on the surface of the nanoparticles.

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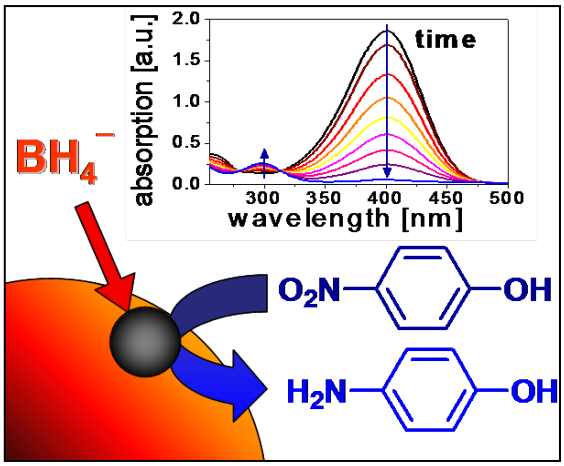
Kinetic Studies of Reduction of 4-Nitrophenol Using Metal Nanoparticles immobilised in Spherical Polyelectrolyte Brushes as Catalyst

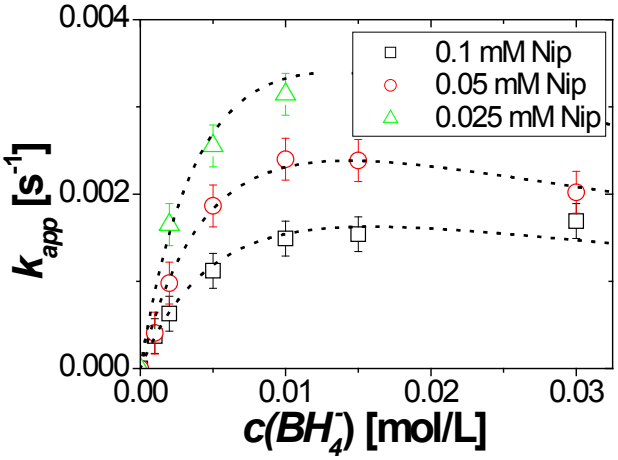
Stefanie Wunder, Yan Lu



Metallic nanoparticles (NP) have been the subject of intense research during the recent years because of their potential use in catalysis. A model reaction suitable for this purpose should be well-defined, that is, no by-products should be formed. Moreover, the degree of conversion should be easily monitored by a simple and fast technique. Therefore the catalytic reduction of 4-nitrophenol (Nip) to 4-aminophenol using sodium borohydride (BH_4^-) as reducing agent has been chosen. The kinetic of the reaction can be analyzed by UV-vis spectroscopy. From the time-dependency of the adsorption peak of Nip at 400 nm the apparent rate constant k_{app} can be calculated.

The proposed reaction mechanism comprise, that both educts must adsorb onto the surface of the metallic nanoparticles. This mechanism is also called the Langmuir-Hinshelwood model:

$$k_{app} = \frac{k \cdot S \cdot K_{Nip}^{n} \cdot c_{Nip}^{n-1} \cdot \left(K_{BH_4} \cdot c_{BH_4}\right)^{m}}{\left(1 + \left(K_{Nip} \cdot c_{Nip}\right)^{n} + \left(K_{BH_4} \cdot c_{BH_4}\right)^{m}\right)^{2}}$$



Furthermore by varying the concentrations of the educts (Nip and BH_4^-) and applying the Langmuir-Hinshelwood reaction model, the reaction rate k and the adsorption constants of 4-nitrophenol K_{Nip} and sodium borohydride K_{BH4}, can be determined for different metallic nanoparticles, respectively. Another measured variable of this model reaction is the induction time. We could show that it is highly dependent on the concentration of Nip but not on that of BH_4^-. Hence, we assume that first a surface restructuring process must occur, before the reaction can start. Further investigation will be focuses on the influence of temperatures on the reaction kinetics in order to get activation energy and adsorption enthalpy by using the van't Hoff equation of the reaction. In addition, it will be also interesting to combine this part of work with theoretic simulation.

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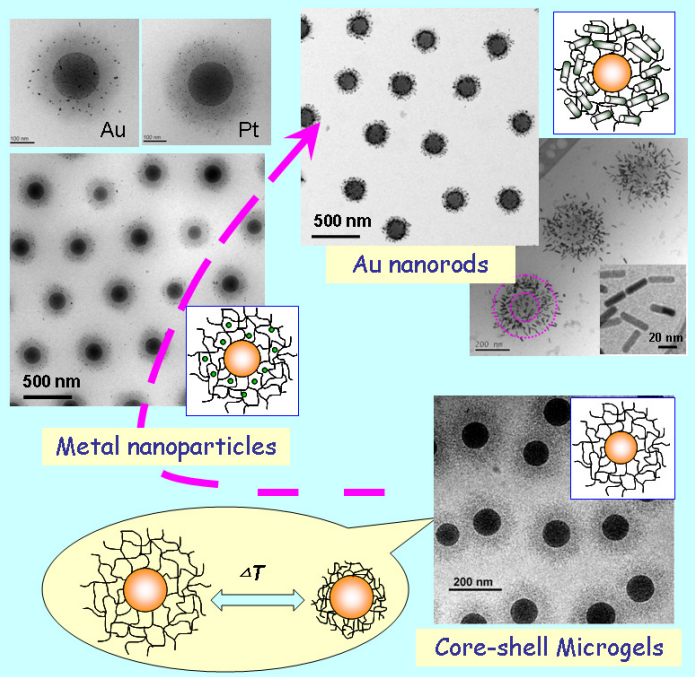
Thermosensitive Core-Shell Microgels as Active "Nanoreactors" for Metal Nanoparticles

Yan Lu



Metal nanoparticles have attracted much attention because such particles may exhibit properties differing strongly from those of the bulk metal. In principle, suitable colloidal carrier system may be used as a "nanoreactor", in which the metal nanoparticles can be immobilized and used for the purpose at hand. The use of microgel particles as reactors for the deposition of metal nanoparticles may have several important advantages over other systems, namely, stability, ease of synthesis and easy functionalization providing stimulus-responsive behavior.

Thermosensitive core-shell microgel particles, in which the core consists of poly (styrene) (PS) whereas the shell consists of poly (N-isopropylacrylamide) (PNIPA) network crosslinked by N, N'-methylenebisacrylamide (BIS), can be used as "nanoreactors" for the deposition of catalytically active metal nanoparticles. Different metal nanoparticles (such as Ag, Au, Pd, Pt and Rh) as well as Au nanorods can be homogeneously embedded into the microgel particles.

The catalytic activity as well as the optical property of metal nanoparticles immobilized in thermosensitive microgels can be tuned by the volume transition within the microgel. At low temperatures, the composite particles are suspended in water, which swells the thermosensitive network attached to the surface of the core particles. At higher temperatures (T > 32°C), the PNIPA-network, however, undergoes a volume transition, in which most of the water is expelled. We demonstrate that the catalytic activity of the microgel-metal nanocomposites can be tuned by the volume transition within the microgel of these systems by using the catalytic reduction of 4-nitrophenol as the model reaction. Thus, the microgel particles present an "active" carrier system for applications in catalysis.

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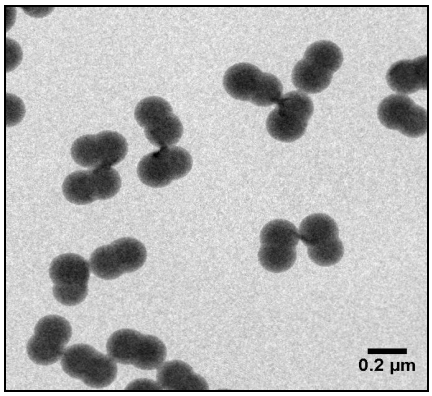
[5] S. Wu, J. Dzubiella, J. Kaiser, M. Drechsler, X. Guo, M. Ballauff, Y. Lu, *Angew. Chem.* **2012**, *DOI: 10.1002/anie.201106515*.

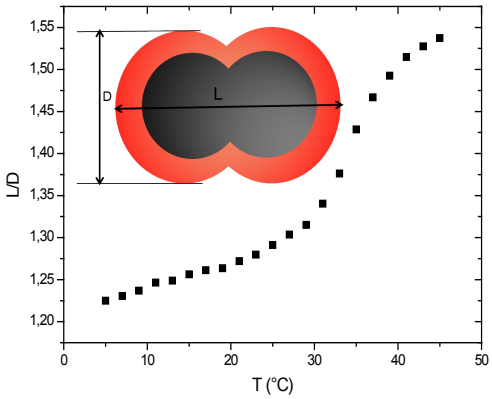
Dynamics of Colloidal Suspensions of Anisometric Particles

Fangfang Chu, Yan Lu, Joachim Dzubiella

Colloid and nanoparticle science has traditionally been focused on dynamics and structural arrest of spherical nanoparticles. However, anisometric particles have attracted more and more attention as the anisotropy in shape is expected to modify the dynamics and equilibrium structures. To investigate the mechanical properties, aqueous dumbbell-shaped particles (DSP) consisting of the polystyrene (PS) core and a thermosensitive cross-linked poly (N-isopropylacrylamide) (PNIPAM) shell are prepared.



TEM image of dumbbell-shaped particles.



Aspect ratio of DSP as a function of temperature.

Despite that several strategies are available to synthesize the dumbbell-shaped particles with the size ranging from 100 nm to several microns, it is still hard to control the particle morphology and to conduct synthesis in large portion, which is, however, essential for potential applications. To solve these problems, homogeneous dumbbell-shaped particles are prepared by seeded emulsion polymerization. In addition, the aspect ratio can be adjusted quantitatively by monitoring the temperature due to the thermosensitive PNIPAM shell. The morphology has been investigated through electron microscopy and light scattering techniques.

Based on the rotational and translational motions of anisometric particles, Näive mode coupling theory (NMCT) predicts that there are two or three dynamic phases: fluid and double glass or fluid, plastic glass and double glass, from which the triple point is expected to be L/D = 1.43. Rheological measurements are planned to investigate the dynamics and the arrest structural of anisometric particles. Combined with theoretical simulations, DSP presents a versatile model system to investigate the fluid-solid transitions of concentrated dispersions of anisometric particles.

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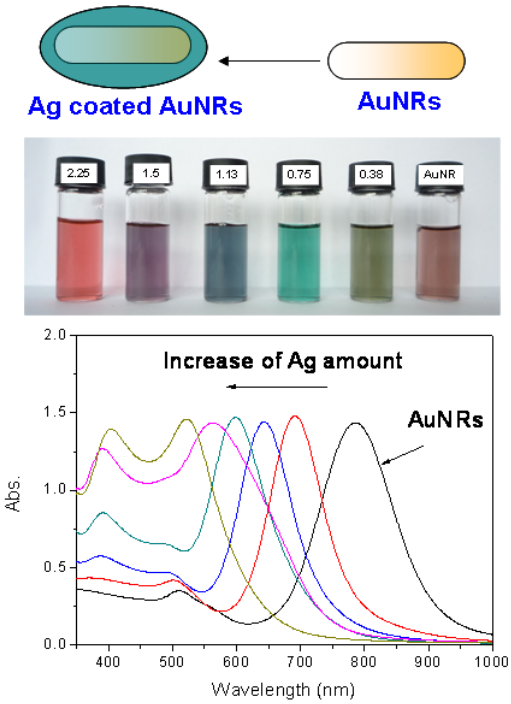
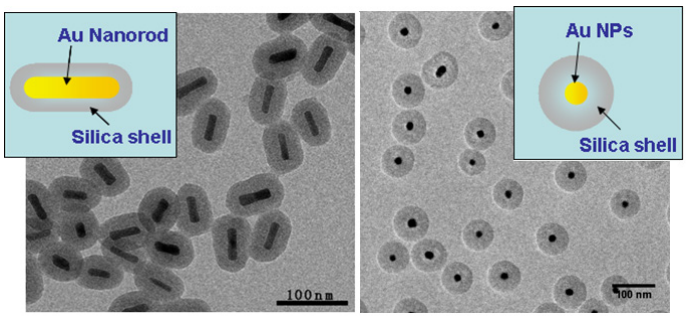
Plasmonic Au – Based Nanocomposite Particles: Synthesis, Characterization and Application

Andreas Ott, Yan Lu, Oliver Benson*

Plasmonics has become one of the most active fields in nanophotonics. In the last several years, there has been a rapid increasing activity within this field as its wide application field ranging from sensing and biomedicine to imaging and information technology. Previous reports have demonstrated that the shape and structure of metal nanocrystals play the most important roles in determining the number, position, and intensity of localized surface plasmon resonance modes.



During our research work, Au and Ag nanocrystals with defined size and shape have been fabricated by the simple wet-chemical method, which is one of favoured routes toward the cost-effective large scale production of metal nanostructures. For example, gold nanorods (AuNRs) can be prepared by seeded-mediated method, which provide two intrinsic (transverse and longitudinal) bands. Deposition of Ag on the AuNRs surface leads to a blue shift in the longitudinal surface plasmon absorption band of Au. Thus, it is possible to prepare nanoparticles with controllable surface plasmon band by this approach.



On the other hand, surface plasmon polaritons (SPP) can guide light and confine it to sub-wavelength dimensions. Recently, evidence for a surface plasmon laser (spaser) was reported. The relatively large loss in the metallic spaser cavity can be compensated by significant gain in a surrounding gain material. Thus, it is crucial to design a metal core/conjugated organic shell hybrid structure that shows coherent amplification of SPP modes and spaser action. For this purpose, metal nanoparticles with different shapes will be synthesized. After covering these metal nanoparticles with a homogeneous SiO_2 layer, organic molecules will be immobilized inside to form the metal core/conjugated organic shell hybrid structure. Here, developing novel metallo-organic nanoparticles is essential not only for the practical applications but also for fundamental understandings.

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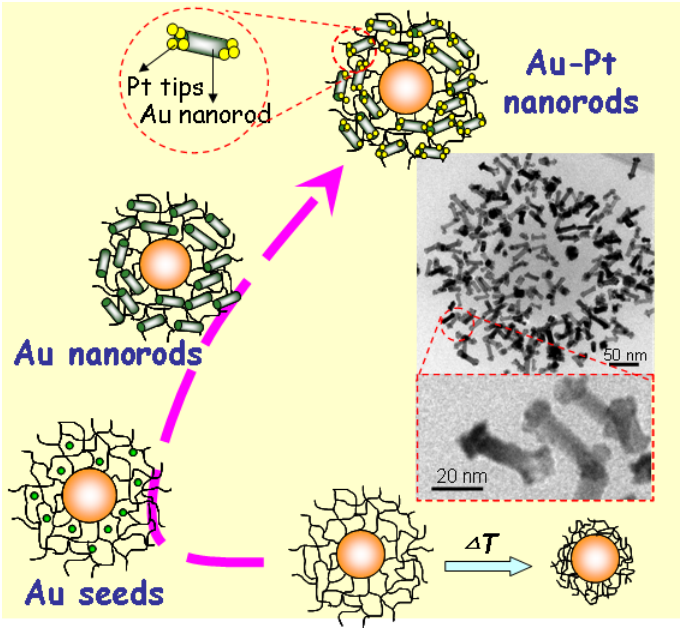
* Humboldt-Universität zu Berlin

In-situ Growth of Catalytic Active Au-Pt Bimetallic Nanorods in Thermo-Responsive Core-Shell Microgels

Yan Lu, J. Yuan*, F. Polzer, M. Drechsler*, J. Preussner+



Bimetallic Au-Pt nanorods (NRs) can be *in-situ* grown into thermosensitive core-shell microgel particles by a novel two-step approach. This demonstrates for the first time that the control of the shape as well as hierarchical structure of nanoparticles in the microgel particles is achievable via an *in situ* strategy. The volume transition of microgel network leads to a strong red shift of the longitudinal plasmon band of the Au NRs, which are immobilized in microgel networks. Platinum can preferentially deposit onto the tips of Au NRs to form dumbbell-shaped bimetallic nanoparticles. The novel synthesis forms bimetallic Au-Pt NRs immobilized in microgels without impeding their colloidal stability. Quantitative analysis of the catalytic activity for the catalytic reduction of 4-nitrophenol indicates that bimetallic Au-Pt NRs show highly enhanced catalytic activity, which is due to the synergistic effect of bimetallic nanoparticles. The catalytic activity of immobilized Au-Pt NRs can be modulated by the volume transition of thermosensitive microgels. This demonstrates that core-shell microgels are capable of serving as “smart nanoreactors” for the catalytic active bimetallic nanoparticles with controlled morphology and high colloidal stability.

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[3] Y. Lu, S. Proch, M. Schrinner, M. Drechsler, R. Kempe, M. Ballauff, *J. Mater. Chem.* **2009**, 19, 3955.
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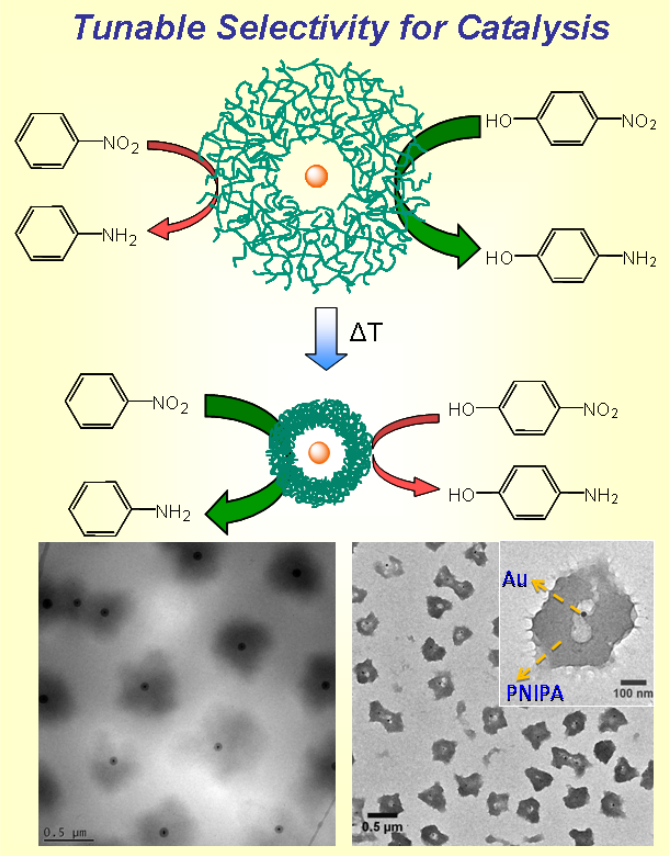
* Makromolekulare Chemie II, University of Bayreuth

+ Metallische Werkstoffe, University of Bayreuth

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



We introduce an inorganic-organic hybrid yolk-shell nanostructure that contains metallic Au nanoparticle as core and thermosensitive microgel poly(N-isopropyl-acrylamide) (PNIPA) as shell. The porosity and the hydrophobicity of this shell can be tuned in a well-defined manner by temperature while the colloidal stability of the entire hybrid is fully maintained. The catalytic selectivity of Au-PNIPA yolk-shell nanoparticles can be tuned between hydrophilic and more hydrophobic molecules through the volume transition of PNIPA shell for the catalytic reduction of 4-nitrophenol (4-NP) and nitrobenzene (NB) in the presence of $NaBH_4$. It is found that the reaction is diffusion-controlled due to the slow diffusion of the reactants through the gel network and concomitant solvation barriers which are very different for the more hydrophobic NB and hydrophilic 4-NP. Temperature can be used as a trigger to enhance the selectivity of the catalysis for a given substrate: 4-NP reacts much faster at low temperature while the reduction of NB is preferred at higher temperature. This selectivity is even enhanced in mixtures of 4-NP and NB. Hence, yolk-shell systems have a great potential to tailor the catalytic activity and selectivity of metal nanoparticles toward a given reaction.

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* State-Key Laboratory of Chemical Engineering, East China University of Science and Technology
[+] Makromolekulare Chemie II, University of Bayreuth

X-Ray Microscopy

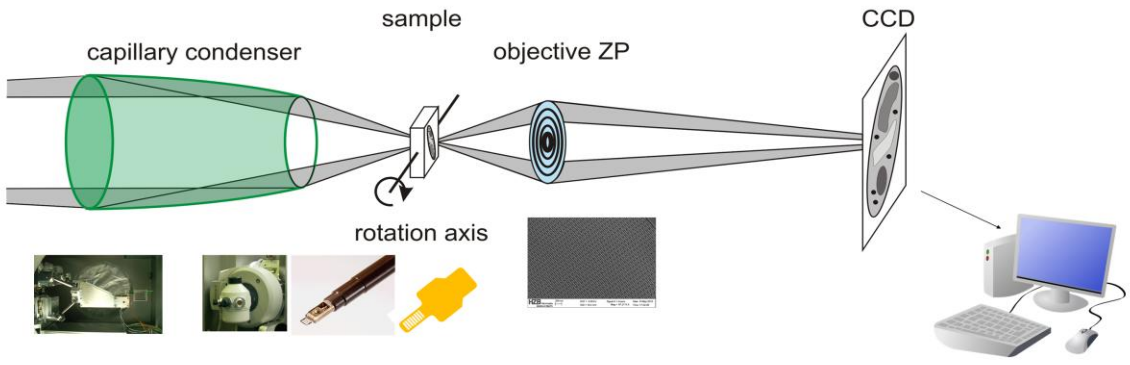
Gerd Schneider

The research work in the Microscopy team comprises the development of advanced optical methods and instrumentation for nanoscale X-ray photonics. The group developed and operates one of the most advanced X-ray microscopes worldwide which permits NEXAFS spectromicroscopy on nanometer length scales with high energy resolution in the soft X-ray photon energy range. In addition, it is the first of its kind which permits high resolution tomography of mammalian cells using their native absorption contrast provided mainly by carbon and oxygen.

The Microscopy group is also one of the world's leaders in the development of high resolution diffractive X-ray optics. With the help of a state-of-the-art electron beam lithography system, special efforts are made towards sub-10 nm focusing. These experimental developments are complemented by theoretical studies based on rigorous coupled wave theory of the volume diffraction of high aspect ratio Fresnel zone plates. In-house developments include also new methods for advanced phase contrast methods combined with 3-D techniques.

Scientific applications comprise materials, environmental and life science. In materials science our main focus is the stress-induced migration and electromigration inside advanced integrated circuit structures. These structures are on the 100 nm length scale and need to be buried inside dielectrics. The X-ray microscope permits due to the high penetration depth of X-rays the imaging of much thicker samples than the TEM which opens up new experiments. These studies are performed in cooperation with industry and the Fraunhofer IZFP Dresden.

In life science our scientific focus is the cell nucleus which is a true 3-D nanoscale structure. Cryo TEM gives us only images of thin sections whereas the cryo full-field X-ray microscope yields the complete cell including the nucleus with its three-dimensional structures like the nuclear membrane channels. In the futures, we want to develop methods for the correlation of fluorescence microscopy and 3-D tomography to provide a widely applicable technique to combine the biochemical information provided by labeled biomolecules in cells with structural information obtained by nano-tomography.



Coworkers:

Dr. Peter Guttmann
Dr. Stefan Rehbein
Dr. Stephan Werner
Dr. Katja Henzler
Dipl. Phys. Basel Tarek

Selected Publications in 2009 - 2012:

1. S. Rehbein, S. Heim, P. Guttmann, S. Werner, G. Schneider, “Ultrahigh-resolution soft x-ray microscopy with zone plates in high orders of diffraction”, Phys. Rev. Lett. 103, 110801 (2009)
2. José L. Carrascosa, Francisco Javier Chichón, Eva Pereiro, María Josefa Rodríguez, Jose Jesús Fernández, Mariano Esteban, Stefan Heim, Peter Guttmann, Gerd Schneider, “Cryo-x-ray tomography of Vaccinia Virus membranes and inner compartments”, J. Struct. Biol. 168 (2009), 234-239
3. Ehrenfried Zschech, Rene Huebner, Dmytro Chumakov, Oliver Aubel, Daniel Friedrich, Peter Guttmann, Stefan Heim, Gerd Schneider, “Stress-induced phenomena in nanosized copper interconnect structures studied by x-ray and electron microscopy”, J. Appl. Phys. 106, 093711 (2009)
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8. S. Werner, S. Rehbein, P. Guttmann, S. Heim, G. Schneider, “Towards high diffraction efficiency zone plates for X-ray microscopy”, Microelectron. Eng. 87 (2010), 1557-1560
9. P. Guttmann, C. Bittencourt, S. Rehbein, P. Umek, X. Ke, G. Van Tendeloo, C. P. Ewels and G. Schneider: “Nanoscale spectroscopy with polarized X-rays by NEXAFS-TXM”, Nature Photonics 6 (2012), 25-29
10. W.G. Müller, J.B. Heymann, K. Nagashima, P. Guttmann, S. Werner, S. Rehbein, G. Schneider, J.G. McNally:“Towards an atlas of mammalian cell ultrastructure by cryo soft X-ray tomography”, J. Struct. Biol. 177 (2012), 179-192
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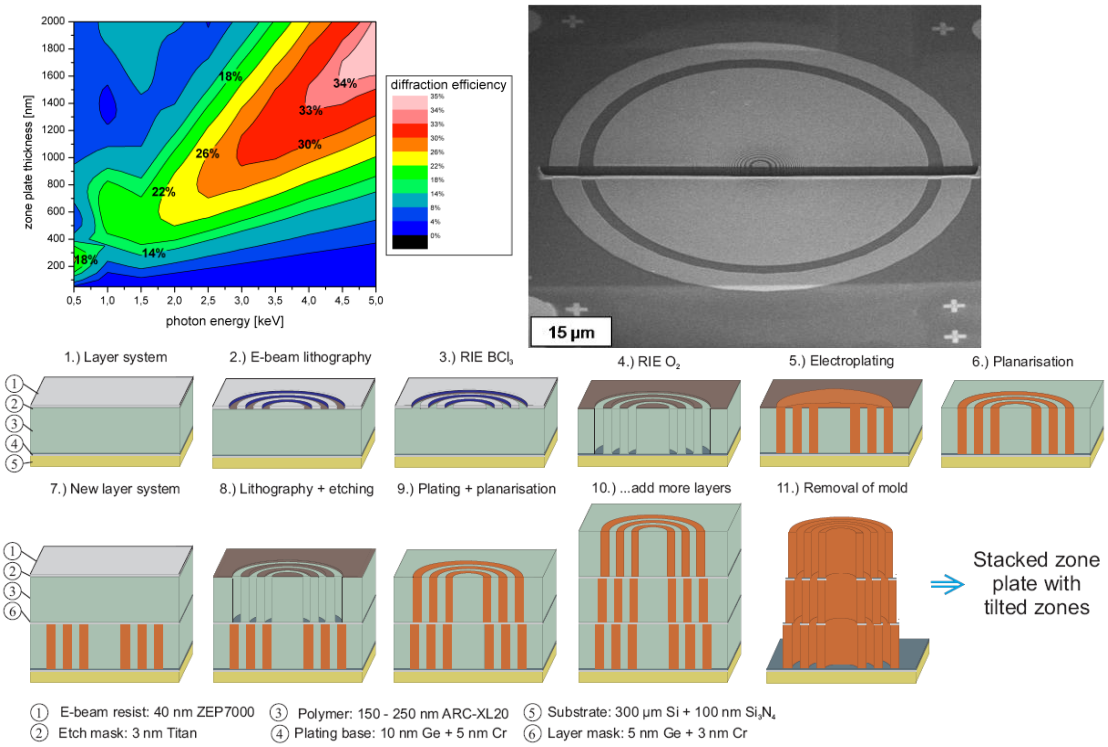
12. G. Schneider, P. Guttmann, S. Rehbein, S. Werner, R. Follath: „Cryo X-ray microscope with flat sample geometry for correlative fluorescence and nanoscale tomographic imaging”, J. Struct. Biol. 177 (2012), 212-223
13. S. Rehbein, P. Guttmann, S. Werner, G. Schneider, “Characterization of the resolving power and contrast transfer function of a transmission X-ray microscope with partially coherent illumination”, Optics Express 20 (2012), 5830-5839

Development of Diffractive Optics for High-Resolution X-Ray Imaging

S. Werner, S. Rehbein, G. Schneider

In the last decade advances in nanostructuring technology lead to rapid progress of the diffractive X-ray optics quality; they became the key elements for high resolution and energy resolving X-ray imaging techniques performed at synchrotron sources. The performance of Fresnel zone plates is characterized mainly by two parameters: The outermost zone width determines the numerical aperture and the height of the zone profile their diffraction efficiency. In the soft X-ray region the ratio of the zone height to the zone width is about 10:1. As sub-25 nm resolution optics are not commercially available, the microscopy group uses the VISTEC e-beam writer for pattern generation and in-house nanotechnology to manufacture these X-ray lenses. For manufacturing zone plates, different steps as thin layer technology, reactive ion etching and electroplating are required. Currently, the resolution obtainable with the HZB zone plates is about 10 nm. Electrodynamic calculations predict that in the future advanced stacked zone pates can combine high resolving power in the sub-10 nm range and high efficiency. High resolution objectives providing a small depth of focusing in the 100 nm range open the way towards optical sectioning. The development of such microscopes for nanoscale 3D imaging strongly depends on stacked zone plates.



[1] S. Rehbein, S. Heim, P. Guttmann, S. Werner, G. Schneider, Phys. Rev. Lett. **103**, (2009) 110801

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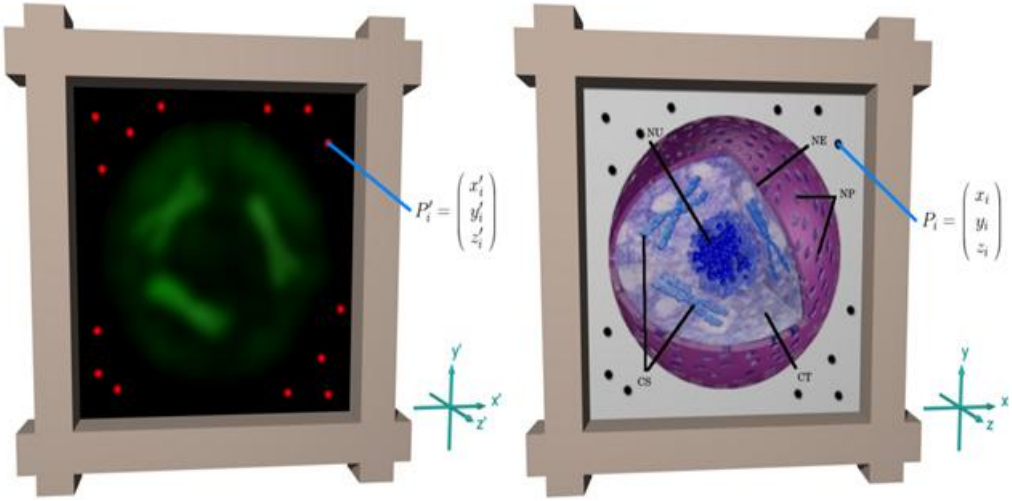
Correlative 3-D Microscopy for Life Sciences

P. Guttmann, G. Schneider

Fluorescence microscopy is an established technique in biophysical investigations of cells and cell nuclei, whereas 3-D X-ray microscopy is a relatively new approach with great potential which enables imaging of whole hydrated cells without chemical fixation, drying or slicing techniques as required in electron microscopy. Conventional optical fluorescence images are diffraction-limited to ~200 nm, whereas current X-ray images can achieve a ten-fold improvement in resolution. The interaction of X-rays is element specific; therefore, X-ray nano-tomography can be used to quantify the packing density of organic material. However, different proteins or molecular structures cannot be distinguished directly in X-ray microscope images. This problem is solved by the availability of specific fluorescent probes detectable by fluorescence microscopy. Thus the two imaging modalities are complementary. Since fluorescence and X-ray microscopy permit analysis of whole cells, it is possible to investigate the same cell in both microscopes.



The fluorescence microscope image (left) will provide information about the location of the labeled structures in the cell nucleus and the position (x_i', y_i', z_i') of the markers (fluorescent dots outside the cell). The tomographic reconstruction obtained from the data acquired with the X-ray microscope reveals the internal nuclear structures (e.g., chromosomes, nucleolus, nuclear envelope, nuclear pores, and chromatin). These correlative studies are ideally suited for X-ray microscopy because of its ability to image whole cells in 3-D. This enables high throughput imaging of structures larger than a few hundred nanometer, which would otherwise be extremely time-consuming to locate and then serially reconstruct using correlative fluorescence and cryo electron microscopy of thin cell sections. With correlative microscopy, we expect to develop a widely applicable technique that will yield significant new insights.

[1] G. Schneider, P. Guttmann, S. Heim, S. Rehbein, F. Mueller, K. Nagashima, J.B. Heymann, W.G. Müller, J.G. McNally, Nature Methods **7** (2010), 985-987

[2] G. Schneider, P. Guttmann, S. Rehbein, S. Werner, R. Follath, J. Struct. Biol. 177 (2012), 212-223

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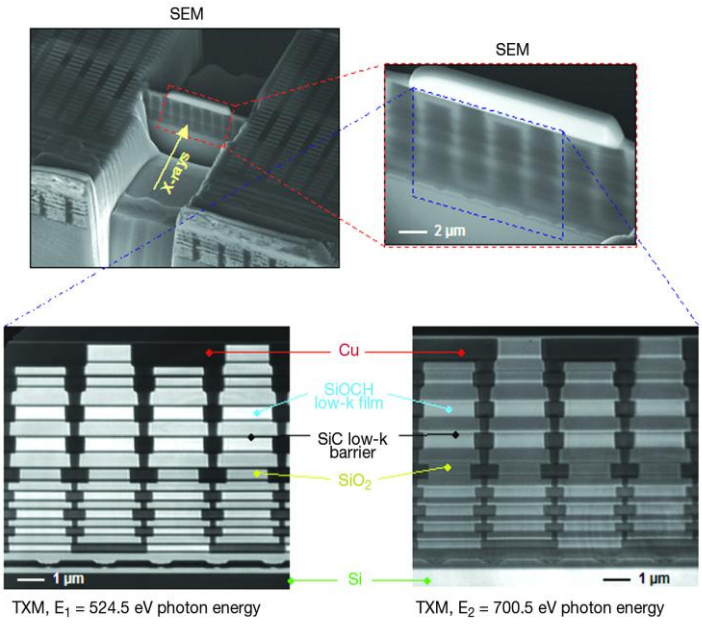
Materials Science Applications

P. Guttmann, S. Rehbein, S. Werner, G. Schneider, E. Zschech*

High-resolution X-ray imaging with a spatial resolution in the 10 - 30 nm range offers unique capabilities for process development and failure analysis in semiconductor industry. Buried metal interconnect structures like copper on-chip interconnects and through silicon vias (TSV) for 3D IC integration can be studied with excellent element-specific contrast. In addition, X-ray nano-tomography permits to study the kinetics of reliability-limiting processes like electromigration (EM) or stress-induced voiding (SIV).

X-ray microscopy is superior to SEM imaging if the structures are embedded as required for in-situ experiments. Such in-situ TXM experiments have been performed that give the ability to visualize mass transport processes and interconnect degradation while stressing fully embedded copper via/line test structures, applying accelerated test conditions (high temperature, high current density). These experiments allow to understand weaknesses in the interconnect technology that cause reliability-related failures. Such real-time imaging of interconnect degradation processes like electromigration and stress migration provide an ability to forecast the effect of process and materials changes on interconnect reliability and to optimize interconnect design rules. Particularly, void evolution in interconnects can be shown for Cu/low-κ structures with high spatial resolution, and rapid pathways for the directed mass transport and weak interfaces can be identified.



SEM micrograph (upper left) showing the FIB prepared fully passivated copper interconnect structures. Due to inelastic electron scattering in the passivation layer, the resolution in the magnified SEM micrograph (upper right) is limited. Images were taken at 525.5 eV and 700.5 eV energy using photons that traversed the prepared lamella in the X-ray microscope. Due to the different absorption properties of the used dielectric materials, these X-ray images reveal the different dielectric layers and the copper interconnects with a lateral resolution of about 20 nm.

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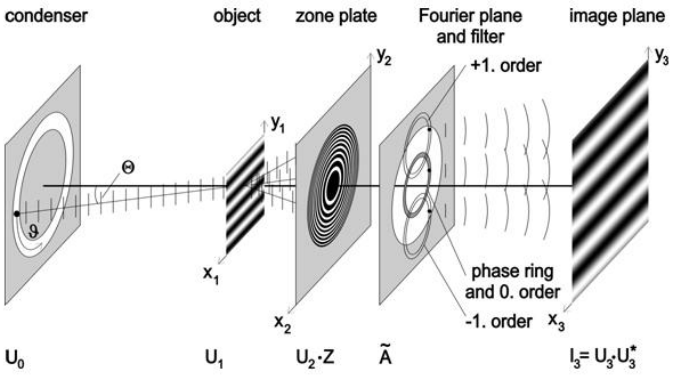
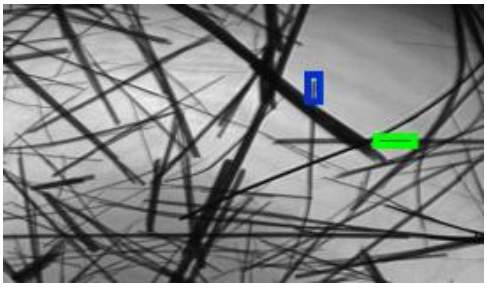
* Fraunhofer-Institut für Zerstörungsfreie Prüfverfahren (IZFP-D), Dresden (IZFP-D)

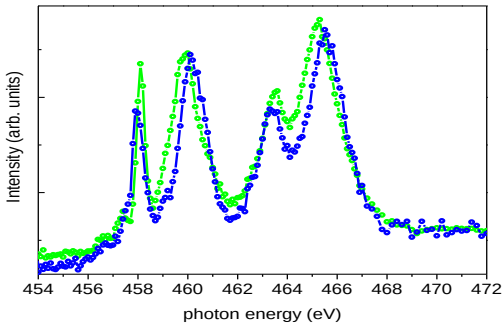
X-ray Optical Methods and Instrumentation for Advanced X-ray Microscopy

G. Schneider, P. Guttmann, S. Rehbein, S. Werner

In the nano-ages new tools for the analysis of complex structures is essential. So far the nano-world was mainly inspected by electron microscopy using a variety of different methods to utilize the image contrast formed by the interaction of electrons with matter. X-ray imaging methods are relatively new and much less developed compared to traditional microscopy techniques. However, they provide at least the same variety of interactions with matter to detect specific elements or chemical bonds. All this is based on high quality X-ray optics and advanced X-ray optical setups which take into account the relatively low efficiency of X-ray optics. Therefore, one goal of the X-ray microscope group is the development of novel methods for X-ray imaging to make use out of the unique interactions of X-rays with matter. X-ray optical setups providing a high energy resolution are required for spatially-resolved NEXAFS, special illumination schemes need to be developed for high-resolution phase contrast imaging or small focal spots using spatially coherent X-ray beams are required for nano-focusing. In addition, for the new instruments and applications special sample environments are required, for example cryo temperatures. Investigations of functionalized samples need linear or circular polarized light to visualize their inherent properties (e.g. polarization dependence of NEXAFS spectra of TiO_x nanoribbons as shown in the figure). To overcome the 3D resolution limitation of currently 30 - 60 nm by the decreasing depth of focus in nanoscale tomography, we work towards a confocal STXM to obtain optical sections with spatial resolutions on the order of 10 nm x 10 nm x 100 nm in x,y,z-direction.







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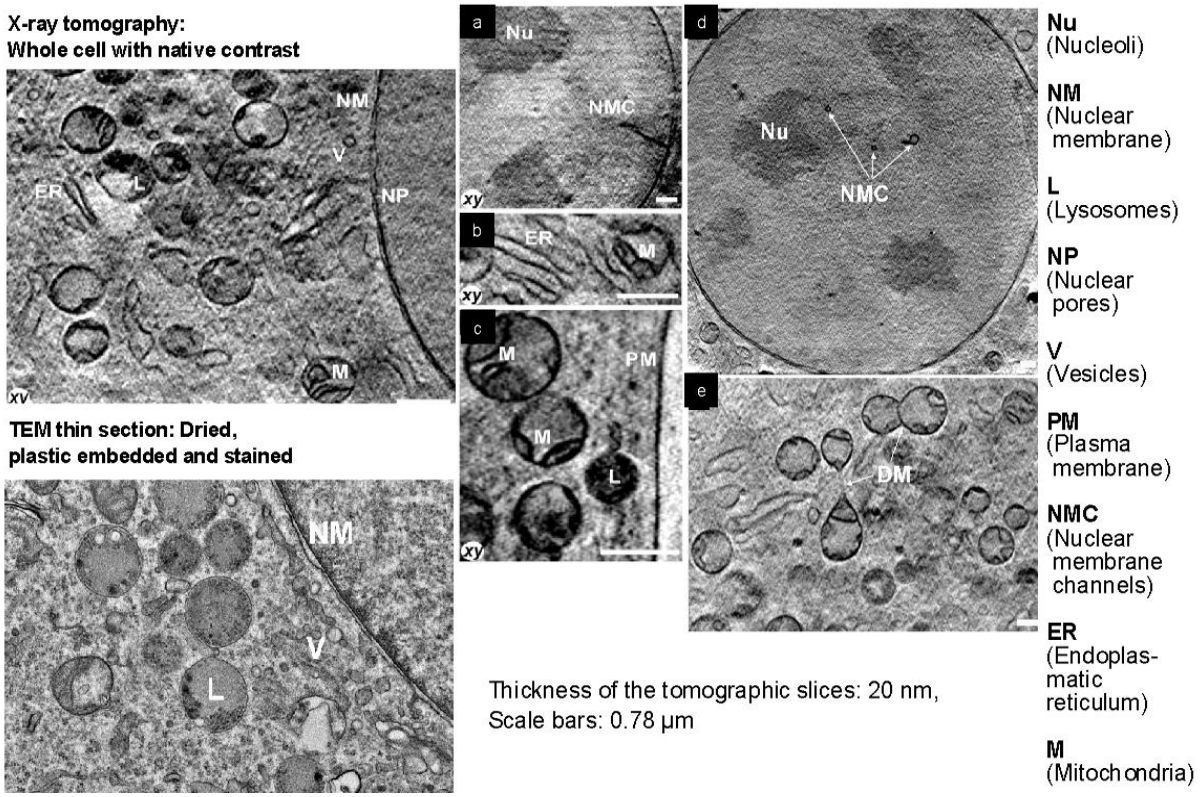
Nano-Tomography of Cells

G. Schneider, P. Guttmann, S. Werner

Soft X-ray microscopy allows obtaining nano-scale 3D images of intact cells using only the natural contrast afforded by the different absorption or phase shift of organic matter and water. This permits entire cells to be examined while in their native state without chemical fixation, chemical staining or physical sectioning, but with only cryo-preservation.

Currently, the only alternative for visualizing 3D mammalian cell ultrastructure is cryo electron tomography performed on multiple serial sections, each of a thickness of much less than a micron, which must then be aligned to produce a 3D image. This is a painstaking process that requires 2-3 weeks per cell. Much faster nano-scale 3D imaging of intact cells can be performed with fluorescence super-resolution microscopy. However, this approach is fundamentally limited to examining the distribution of a few molecular markers per cell, and is therefore incapable of resolving a full spectrum of ultrastructural features. Thus X-ray microscopy with its potential to reveal the 3D ultrastructure in intact cells with a thickness of 10 µm fills an existing gap in current microscopy methods.



The new generation X-ray microscope at HZB allows routinely to visualize the plasma membrane, nuclear membrane, nuclear pores, nucleoli, endoplasmic reticulum, vesicles, lysosomes and mitochondria. It is now also possible to resolve internal organellar structures, such as mitochondrial cristae, the double nuclear membrane and lysosomal inclusions.

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[2] W.G. Müller, J.B. Heymann, K. Nagashima, P. Guttmann, S. Werner, S. Rehbein, G. Schneider, J.G. McNally, J. Struct. Biol. 177 (2012), 179-192

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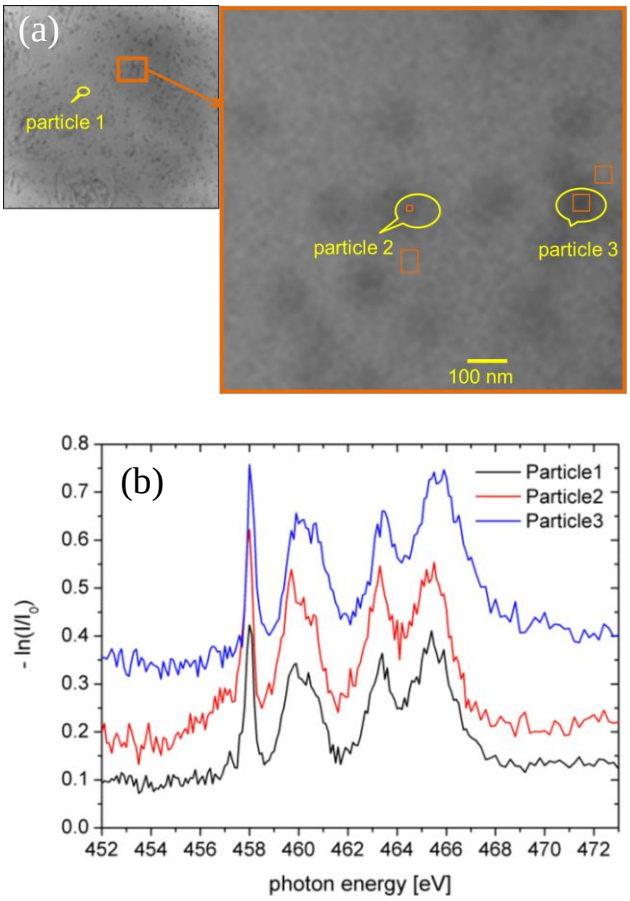
NEXAFS/cryo-TXM on colloidal hybrid structures

Katja Henzler, Peter Guttmann, Yan Lu, Gerd Schneider

The properties of colloidal organic-inorganic hybrid materials are significantly affected by their morphology and elemental composition [1,2]. Therefore, suitable analytical methods are required for the investigation of these systems in situ. In order to investigate the morphology and composition of such particles in their solvated state, full-field cryo transmission X-ray microscopy (TXM) with automated nano-spectroscopy was used [3]. The investigated hybrid particles consist of a spherical polyelectrolyte brushes (SPB) which act as a carrier system for metal or metal oxide nanoparticles [1,2].

In the presented work we investigated titanium dioxide (TiO_2) nanoparticles on SPB by nano-spectroscopy at the Ti L-edge and O K-edge. The TiO_2 nanoparticles were synthesized by a modified sol-gel-process [1,2]. The diameter of these titanium dioxide particles was around 12 nm[1], which is in the resolution range of the HZB cryo-TXM at the BESSY II U41 undulator beamline [3]. The introduced method offers the possibility to distinguish between the different crystal structures of titanium dioxide. This is essential since only nanoparticle with an anatase crystal structure shows a high photolytic activity.



(a) TXM-image of spherical polyelectrolyte brush with titanium dioxide nanoparticles. Highlighted are the particles which were used to record the NEXAFS spectra (b) at the Ti L-edge.

(b) NEXAFS spectra at the Ti L-edge of titanium oxide nanoparticles. The careful analysis of the recorded spectra reveals that the TiO_2 nanoparticles have an anatase crystal structure.

Figure (a) shows a TXM-image between 458 eV- 465.4 eV. Additionally, the used particles for the recording of the NEXAFS spectra at the Ti L-edge (Figure (b)) are highlighted. From the presented NEXAFS spectra in Figure (b) the electronic structure of the titanium dioxide can be calculated. Consequently, the crystal structure of titanium dioxide nanoparticles can be determined. The thoroughly evaluation of the presented spectra in Figure (b) shows that we have indeed an anatase crystal structure. This is in accordance with X-ray diffraction data we have collected on dried samples of this system.

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Soft Matter Theory Group: Methods, Mission, and Research

Joachim Dzubiella

The theory group headed by Prof. J. Dzubiella establishes another important pillar of the Soft Matter and Functional Materials Institute at the HZB by providing theoretical models of soft (biological) condensed matter systems using analytical calculations and computer simulations. The topics studied by the group cover a broad range of systems and include, for instance, the nonequilibrium structure and phase behavior of colloidal and polymeric fluids, protein structure and dynamics in aqueous (poly)electrolyte solutions, or self-organization of soft organic materials at interfaces.

Inherent to any complex soft matter system is the appearance of multiple length and time scales. In order to tackle the multiscale challenge theoretically, the many small (and fast) degrees of freedom need to be integrated out, what the theorists call 'coarse-graining'. The latter can be achieved by combining various theoretical treatments. Here, the theory group employs approaches from classical statistical mechanics, liquid state theory, and various computer simulation techniques. In particular, the group performs Molecular dynamics (MD), Brownian dynamics (BD), or Monte-Carlo (MC) simulations combined with free energy calculations and classical dynamical density functional theory.

The computationally demanding MD simulations of molecular systems are performed on the high performance cluster *Dirac* directly located and maintained on the Lise-Meitner-Campus. The cluster currently features 30 nodes embracing 700 computing processor units (CPUs) and parallel batch processing software and state-of-the art MD computer simulation software. Given the excellent personal and spatial infrastructure at the HZB the cluster warrants competitive computer power for the planned theoretical undertakings at the Soft Matter Institute.

The theory group thus directly complements the experimental efforts in soft matter physics at the HZB. It provides deeper physical insights by modeling and boiling down the complex reality to simple model systems. Atomistically-resolved MD simulations on the other hand enlighten the molecular mechanisms behind certain phenomena inaccessible for direct measurement or spatial resolution. Last but not least, the theory group will support and guide software developments for analyzing and modeling structural data obtained from HZB beamline users. The strong synergy between the theory and experiments concentrated here at the HZB will hopefully result in fascinating discoveries in soft material science in the coming years.

List of coworkers:

Prof. Dr. Joachim Dzubiella
Dr. Jan Heyda
Karol Palczynski (PhD student)
Nils Heptner (PhD student)
Cemil Yigit (PhD student)
Anja Muzdalo (Master student)

Selected publications

1) 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, *Angewandte Chemie*, to be published (2012).

2) Core-shell microgels as "smart" carriers for enzymes
N. Welsch, A. L. Becker, J. Dzubiella, M. Ballauff, *Soft Matter* **8**, 1428 (2012).

3) Effects of Hofmeister ions on the α-helical structure of proteins
A. H. Crevenna, N. Naredi-Rainer, D. C. Lamb, R. Wedlich-Söldner, J. Dzubiella, *Biophys. J.,* to be published (2012).

4) Ionic-specific excluded-volume correlations and solvation forces
I. Kalcher, J. C. F. Schulz, and J. Dzubiella, *Phys. Rev. Lett.* **104**, 097802 (2010).

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P. Setny, Z. Wang, L.-T. Cheng, B. Li, J. A. McCammon, and J. Dzubiella, *Phys. Rev. Lett.* **103**, 187801 (2009).

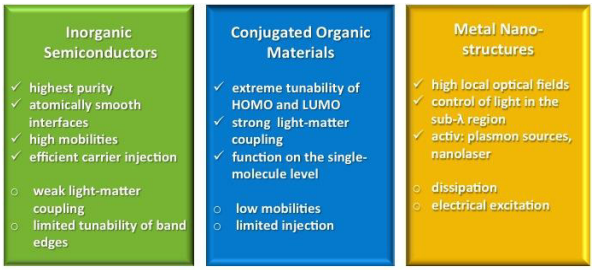
6) Salt-specific stability and denaturation of a short salt-bridge forming alpha-helix
J. Dzubiella, *J. Am. Chem. Soc.* **130**(42), 14000-14007 (2008).

7) Nonequilibrium sedimentation of colloids on the particle scale
P. Royall, J. Dzubiella, M. Schmidt, and A. van Blaaderen, *Phys. Rev. Lett.* **98**, 188304 (2007).

SFB-951 project A1: Exploring molecular-structure formation of hybrid inorganic/organic systems for opto-electronics (HIOS) by all-atom molecular dynamics computer simulations

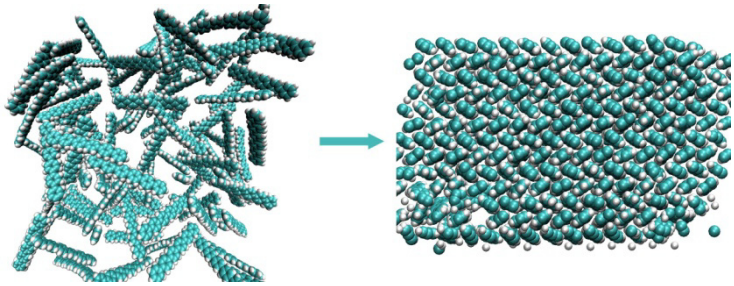
Karol Palczynski

Hybrid Inorganic/Organic Systems (HIOS), heterostructures from different materials, currently revolutionize electronic and optical technology. Our long term goal is the exploration of new materials that combine the strengths of organic and inorganic semiconductors and metals.



Materials of HIOS: ✓ strength ○ weakness

The optical and electronic properties of HIOS strongly depend on the molecular configuration of the conjugated organic molecules (COMs) at the inorganic semiconductor surfaces. The SFB-951 is a DFG-funded Collaborative Research Centre to research HIOS. Within the CRC the goal of project A1 is to explore the nucleation and self-assembly of COMs on ZnO surfaces and in bulk crystals by applying atomistically resolved classical molecular dynamics (MD) computer simulations.



Study of COM bulk structure formation: Crystallization of systems composed of a few hundred COMs can be simulated effectively in a matter of days on a local cluster.

OBJECTIVES

-Study COM bulk structure formation

-Investigate interactions at organic/inorganic interfaces and bulk structure deformation near surfaces

-Find effective COM-COM pair potentials and COM-Surface interactions suitable for coarse graining

METHODS

Molecular dynamics software packages such as GROMACS, coupled with strong computational power, calculate molecular trajectories by integrating Newton's equations of motion employing intramolecular and intermolecular interactions from force-fields adjusted for organic molecules.

We ascertain the relationship between parameters (such as partial charges, Lennard-Jones parameters, temperature, pressure) and the bulk structure of organic molecular crystals composed of Coronene, Diindenoperylene or p-Sexiphenyl and investigate the fundamental interactions governing nucleation of COMs on inorganic surfaces and study structure-dependence on temperature and coverage. Results will be compared to X-ray scattering experiments and equilibrated structures will be proposed for ab-initio calculations.

[1] https://www.physik.hu-berlin.de/hzmo/hios

Modeling protein adsorption using atomistic and coarse-grained computer simulations

Cemil Yigit

The central goal of this theoretical project is the prediction of the thermodynamics of protein absorption into oppositely or like-charge polyelectrolyte (PE) brushes. Because of their amphoteric character (i.e., hydrophobic vs hydrophilic amino acids), proteins have surface active properties, and absorption stems from a sensitive balance of competing energetic and enthalpic interactions. In the experiments at the experimental biophysics group here at the HZB, entropic and enthalpic energies of absorption can be measured with high accuracy [1], but the molecular mechanisms of absorption have remained poorly understood. Our goal is to uncover clearly the detailed mechanisms of protein adsorption by using a multiscale-modeling approach.

In particular, the desired results shall be achieved by different techniques ranging from MD computer simulations, thermodynamic integration, replica exchange methods or 'steered' MD methods. Based on simulations at different temperatures one can compute the entropic and enthalpic contribution to the free energy of absorption, and the molecular origins can be illuminated and identified. This will provide a better understanding of the fundamental physical processes behind protein adsorption.

As an example, figure 1 and 2 show preliminary simulations of (1) lactoglobulin close to a polyelectrolyte brush, and (2) the interaction between a positive patch of lactoglobulin and polyacrylic acid (1), respectively.

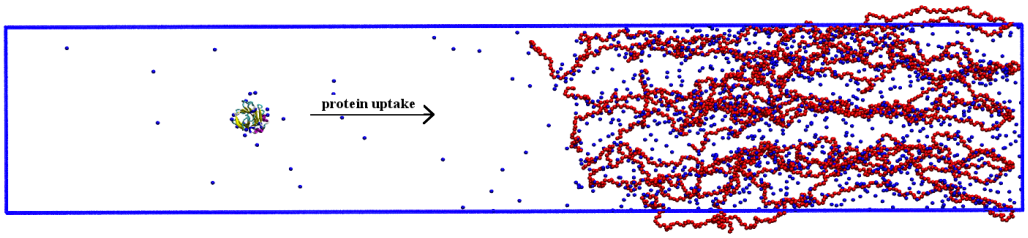


Figure 1: Snapshot of a lactoglobulin protein approaching a planar polyelectrolyte brush taken from a coarse-grained (Brownian dynamics) computer simulations, where water is only implicitly modeled by a dielectric constant. The computational box (blue lines) is periodically repeated perpendicular to the direction of brush extension. 4x4 polyelectrolytes (red chains) are tethered to the right wall.

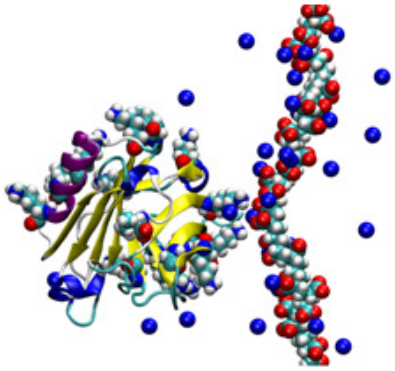


Figure 2: Snapshot of one polyelectrolyte chain (polyacrylic acid) interacting with a positive patch of a lactoglobulin protein from atomistic MD computer simulations. Positively charged atoms (and Na+ counterions) are rendered in blue, negatively charged atoms in red. Water molecules are omitted for clarity.

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Structure and Dynamics of Colloidal Dumbbell Suspensions

Nils Heptner

Non-equilibrium dynamics of colloidal dispersions, i.e. colloids driven by external forces, are of high interest in soft matter physics. This project aims to complement the experimental effort to investigate the structural evolution of colloids. Particularly dynamic phase transitions and pattern formation give rise to exciting questions. In particular we will calculate shear stress, relaxation properties, and dynamical structure factors, which are accessible to experimental workers.

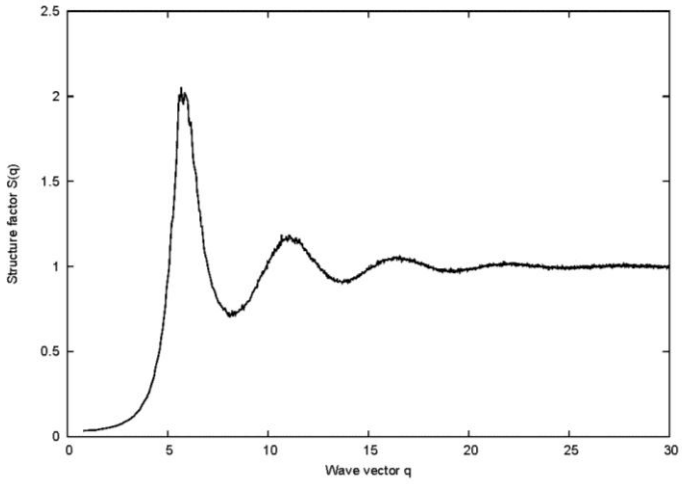

Figure 1: Static structure factor S(q) of dumbbells in fluid phase from BD simulation

One goal of this project is to add to the understanding of the rheology of high volume fraction colloidal dispersions under shear by using methods of theoretical physics. The computational technique of choice is Brownian dynamics (BD). [1] In addition to BD simulations dynamical density functional theory (DDFT) is planned to be applied.[2] In DDFT the evolution of the density is given by

$$\frac{\partial \rho}{\partial t} = \nabla \cdot \left[\rho \nabla \frac{\partial F[\rho]}{\partial \rho} \right],$$

where F is a Functional of the average density $\rho(\vec{r}, t)$.
The influence of hydrodynamic interaction on the Rotne-Prager level will be investigated. Particle geometries of interest will include hard spheres, dumbbells, and chains of colloidal beads. Static and dynamic properties will be calculated from the simulation data in order to add to the interpretation of ongoing experiments at the HZB soft matter institute [3].

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Salt Induced Hydrophobic Collapse of Biopolymers

Jan Heyda

Polymers are key macromolecules for the development of new materials, medical substances, and the function of life. The properties of biological and synthetic polymers in aqueous solution are tightly linked to their conformation, which, in turn, is sensitively controlled by the salt type and concentration in the solvent [1]. Ion-specific effects (or called Hofmeister effects), however, on polymer assembly and hydrophobic collapse are poorly understood. The prediction of salt-specific effects is highly desirable to manipulate and control conformational changes. Very recently, the empirical concept of 'ion partitioning' at biopolymer surfaces was presented as a route towards the free energy change upon biopolymer collapse [2].

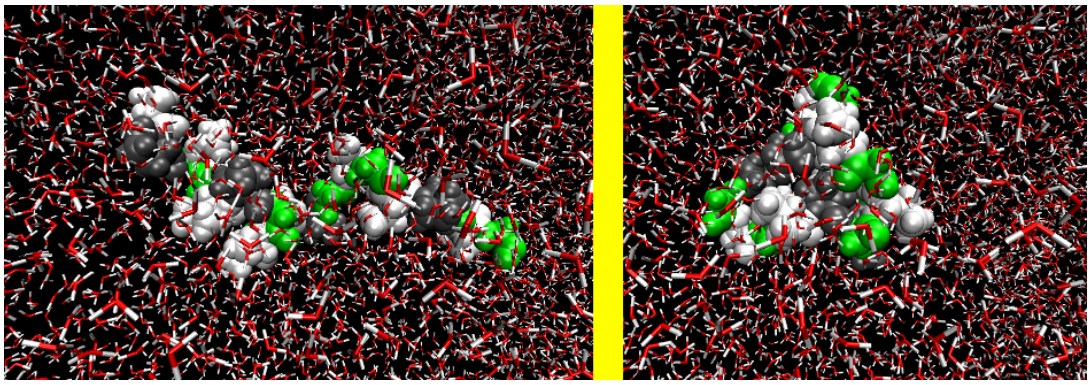

Figure: Structures of elastin like polypeptide (glycine residues in green, proline in black, and valine in white) from REMD calculation. The uncollapsed state at low temperatures (left) and collapsed state at high temperatures (right).

We suggested combining the explicit-water computer simulations and the SASA analysis together with the experimentally derived ion partitioning concept. This enables the prediction of ion-specific effects on the lower critical solution temperature (LCST) in hydrophobic polymer collapse of Poly-N-isopropylacrylamide (PNIPAM), and elastin-like peptides. Both systems are well experimentally characterized.

Apart from use in biotechnological applications and material design, our theoretical framework may help illuminate processes important for polymeric drug delivery agents as well as for medical substances to avoid protein misfolding and aggregation responsible for a large number of human diseases (i.e. Alzheimer disease).

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[2] Pegram, L.M., Wendorff, T., Erdmann, R., Shkel, I., Bellissimo, D., Felitsky, D.J. and Record, M.T. Why Hofmeister effects of many salts favor protein folding but not DNA helix formation. *Proceedings of the National Academy of Sciences of the United States of America*, **2010**, *107*, 7716.

Time-Of-Flight-Spectroscopy

Margarita Russina

Development of new instrumental approaches, pushing the limits of the existing methods and application of neutron spectroscopy in creative ways is a focus in the research activities if the Time-Of-Flight Spectroscopy Group. The group develops and operates Time-Of-Flight Spectrometer NEAT, is engaged in the development of novel instrumentation and methods for European Spallation Source Project and applies time-of-flight neutron spectroscopy to study the relationship between the microscopic dynamics and macroscopic function in disordered and nanostructured materials.

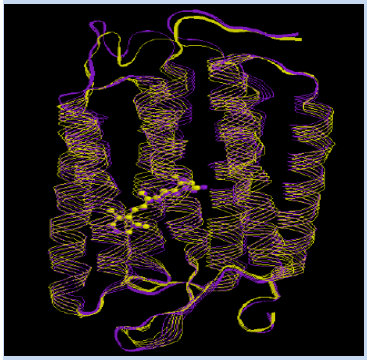
The cold neutron Time-Of-Flight spectrometer NEAT is the key instrument in the HZB neutron instrument suite to study the dynamics and structure on the very broad frequency range (10meV -150meV) and on a length scale from 0.5 to 100 Å . The neutron Time-Of-Flight spectroscopy can be applied to study extremely broad range of phenomena in complex systems, e. g. quantum phenomena in magnetism, microscopy dynamics of proteins, and fast ionic diffusion in conducting systems. NEAT was built between 1987 and 1993 as one of the key instruments funded within the reactor upgrade project. Thanks to its innovative design NEAT delivered one of the highest counting rates worldwide in 1993 despite of the moderate reactor power. The spectrometer was continuously highly demanded by users with 30 experiments served yearly. Further substantial intensity gain and new instrumental capabilities were achieved in 2005 by optimizing the chopper operating parameters and introducing of new sample environments. These measures boosted scientific work on NEAT even more and were followed up by significantly enhanced publication record by both external and internal users in high ranking journals such as Physical Review Letters, J. Physical Chemistry Letters etc. However, new scientific developments dictated the necessity for new instruments with higher power and new capabilities. Therefore full upgrade of the instrument using state-of-the-art components became inevitable. In the face of the strengthening international competition from newer instruments, we have developed a concept of full upgrade of NEAT for 40 fold increase of sensitivity, in addition to the pending improvements of the cold source of BER II. The upgrade will maintain NEAT at the world class level for the decade to come and provide an outstanding experimental tool for research areas of strategic importance to HZB including magnetism, material science, energy and soft matter. The upgrade proposal has been positively reviewed and approved in the competitive project selection of the Helmholtz Association and currently the project realization is in full progress.

The group is also actively participating in the development of the novel techniques and approaches for instruments at both continuous and pulsed sources. The development is pursued in cooperation with European Spallation Source Project and as part of German ESS contribution project funded by BMBF. The highlights include Multispectral Beam Extraction that enhances spectral features by using cold and thermal neutrons simultaneously on the same instrument or pioneering development of multiplexing techniques such as Repetition Rate Multiplication and Frame Multiplication methods which allow us to take control over the pulse parameters to best suit the instruments and individual experiments. NEAT was also used for the development and first realization of the real-time correlation technique TISANE as part of the collaborative effort between various departments at HZB and Institute Laue Langevin. Multispectral Beam Extraction was successfully implemented at HZB as a signature feature of the advanced time-of-flight (TOF) diffractometer EXED. Repetition Rate

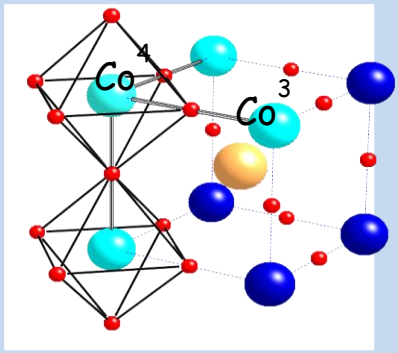
Multiplication is by now realized on instrument LET (ISIS) and a number of the instruments at J-PARC (Japan). Multi-spectral Beam Extraction together with Repetition Rate and Frame Multiplication methods are part of the instrument concept for European Spallation Source.

Important part of the research activities is the study of the dynamic heterogeneities and functionality in disordered and nanostructured materials by help of TOF spectroscopy. The functionality of these systems is often driven by dynamic phenomena happening on the length scale of a few atomic distances. The understanding of these fundamental phenomena is the key to the optimization of these materials to our needs. Our research is focused on the exploration of the nanostructured, energy-related materials and heterogeneous dynamics in proteins. Using neutron scattering complemented by molecular dynamics calculations we were able to discover novel spin based nanoparticles in perovskites, identify mechanisms of ionic conduction in solid electrolytes, observed new quantum-dynamical phenomena of molecular hydrogen caused by the confinement in water clathrates. To support our activities in this area we have established cooperation with Los Alamos National Laboratory, Humboldt University Berlin and with the theory group of F-I2 at HZB.

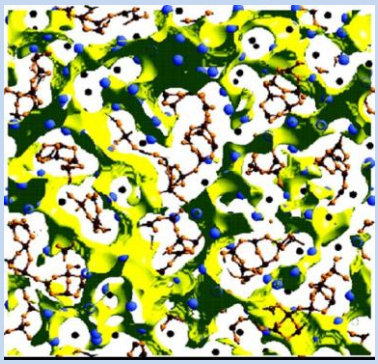
Examples of Neat applications:



Proteins at work



Magnetic nanoparticles



Solid electrolytes

Coworkers

Dr. Margarita Russina
Dr. Nikolaos Tsapatsaris
Dr. Katharina Rolfs
Dr. Gerrit Günther

Selected Publications in 2009-2011

1. M. Russina, Gy. Kali, Zs. Santa, F. Mezei, *Nuclear Instr. and Methods A*, **2011**, doi:10.1016/j.nima.2011.05.077
2. M.Russina, F. Mezei, *Nuclear Instr. and Methods*, A604 (**2009**) 624
3. M. Russina, E. Kemner, M. Cellie, L. Ulivi, F. Mezei, *J. Phys.: Conf. Ser.* 177 (**2009**) 012013.

NEAT Upgrade Project

M. Russina, N. Tsapatsaris, K. Rolfs, M. Ballauff

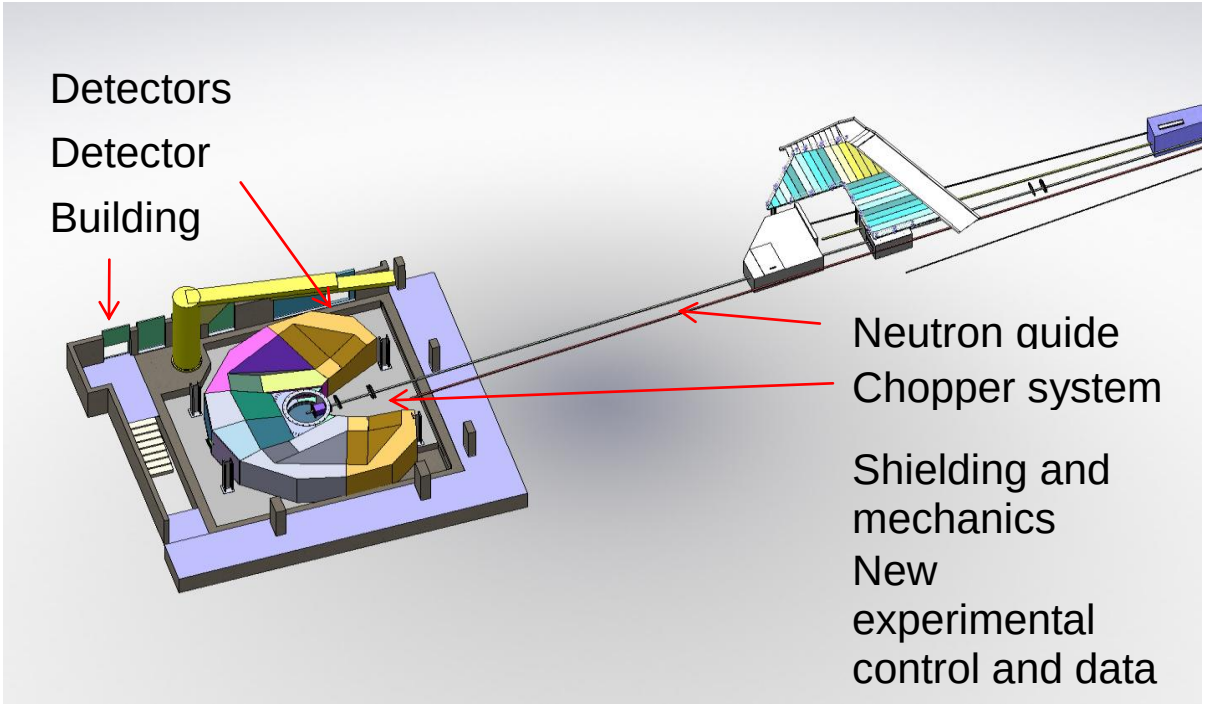
The goal of this proposal is the major upgrade of time-of-flight spectrometer NEAT, which will results in forty-fold intensity gain and development of new instrumental and sample environment capabilities. The new instrument will offer top of the line performance in cold neutron spectroscopy and will provide an outstanding experimental tool by paying special attention to complex and extreme sample environments and in-situ studies for a large spectrum of research areas of strategic importance of Helmholtz Center Berlin including magnetism, material science and soft matter. Further it will join EXED (the unique extreme environment diffractometer at BENSC) as a complementary extreme environment spectrometer further strengthening the profile of BENSC in neutron scattering studies under extreme conditions.
Cold neutron spectroscopy provides a unique and outstandingly versatile tool for exploring microscopic processes. It delivers direct, microscopic information in both space and time, while most other spectroscopic probes are either local (such as hyperfine field methods) or macroscopic in the spatial dimension (such as light scattering). Direct geometry time-of-flight spectrometers are of particular importance in this type of techniques: they can access the dynamical response in very broad time and space domain, and thus offer the most flexible tool for microscopic and nano-scale investigations into complex systems and novel materials. The cold neutron chopper spectrometer NEAT is best suited to probe dynamic phenomena directly in space and time in the time domain 10^{-13} – 10^{-10} s and on the length scale ranging from microscopic (0.5 Å) to nanoscale dimensions (up to about 50 nm). The examples of current NEAT applications are the study structure-dynamics relationship in glasses, nanostructured materials, polymers, proteins, confined matter, molecular magnets, magnetic excitations, spin polarons.

The upgrade of NEAT incorporates:

Full upgrade of primary spectrometer consisting of an advanced neutron delivery system and a new chopper system;
Full upgrade of the secondary spectrometer in order to increase the solid angle coverage with the use of state-of-the art linear position sensitive detectors (PSD). It will require a novel detector chamber with almost 2π coverage in the horizontal scattering plane and a non-magnetic environment for high magnetic fields. The use of PSD will open up the opportunity to use NEAT for study of the single crystals with high data collection speed.
New building to accommodate the new instrument;

As part of the BENSC upgrade program BENSC is planning to install a new cold neutron source and the new beam extraction with much more efficient supermirror guides. The cold source replacement itself results in a factor of around 1.6 increase in cold neutron flux. With the new cold source to be installed in 2011, the upgraded beam delivery neutron optics and chopper system and the much enhanced detector area will lead to intensity gains of more than a factor of 40. The upgraded instrument will therefore open up new fields of applications such as full three-dimensional reciprocal space mapping of excitations in single crystals, thin films and nanoparticles or parametric, in-situ and time-resolved studies of chemical processes. The fully upgraded NEAT with the novel secondary spectrometer cannot be accommodated within the existing neutron guide hall and requires a new extension of the guide hall by a small building, where the new NEAT will be installed. At this new position it

will also be possible to accommodate the 3 m high PSDs and reach the high detector coverage over nearly 2π of the upgraded NEAT. In addition this relocation opens up space for additional instruments and a whole new guide giving ample scope for future developments of innovative instrumentation at BENSC.
The cost of the project is about 9 M€ and it will be completed in 2014. The upgrade of NEAT will decisively strengthen BENSC in the key areas of magnetism, biological- and soft matter and material science research. It will also allow an optimum implementation of the BENSC Upgrade Programme and overall underwrite BENSC's future as an innovative and top of the line facility for the coming decade.



Multiplexing technique: adjustable short pulses at long pulse spallation source

M. Russina

The main challenge in the instrument design at the future European Spallation Source is how to take full advantage of the high neutron peak flux and at the same time to take control over the instrument parameters. Novel Multiplexing techniques, such as Repetition Rate Multiplication [1] and Wavelength Frame Multiplication [2] allow us to create instead of one long pulse a number of mini-pulses with variable frequencies and pulse lengths but with the same peak flux as original long pulse. The underlying principle of multiplexing techniques is to use a set of monochromatic wavelengths or a set of wavelength bands coming from the same source pulse by means of mechanical chopper systems. In this case the instrumental parameters, such as wavelength resolution, wavelength band, repetition rate are not any more determined by the source pulse parameters, but can be flexibly defined by the chopper frequency, speed and chopper pulse.

The design of chopper systems for realization of multiplexing methods is different from those at reactors. Similarly to the reactor based instruments the main requirement for multiplexing chopper system is to assure that a neutron detected at any given time can only come from a single pulse shaping chopper pulse. Multiplexing entails novel features such as “quantized” chopper configurations in terms of the choice of the chopper positions L_i and chopper frequencies fi relative to the source and source frequency f, respectively.

The results of first experimental implementation of Repetition Rate Multiplication (RRM) and Wavelength Frame Multiplication (WFM) methods deliver full proof-of-principle of these methods and demonstrate multiply enhanced data collection rates and individual tuning of the pulse length and/or pulse repetition rate largely independently of the actual source pulse parameters. For implementation we used reactor based TOF spectrometer NEAT at HZB, Berlin [3] and TOF diffractometer at BNC, Budapest [4], operating in non-standard modes. The pulsed source in these studies has been emulated either by a disc chopper rotating with 20-40 Hz or defined by the chopper system as a virtual pulsed source operating around 10 Hz.

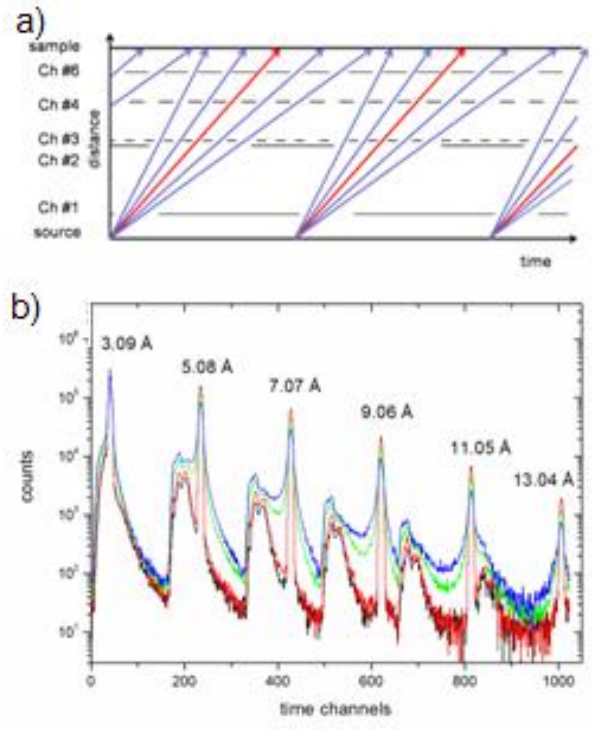


Fig.1, (a) Principle of Repetition Rate Multiplication (RRM) as realized by a disc chopper system and compared to the conventional way of operation using only one monochromatic wavelength (indicated by red arrows in the figure)
(b) Temperature dependent inelastic spectra from water sample confined into porous silica as directly measured using NEAT in the RRM mode at 165.67 Hz pulse repetition rate at the sample and 23.67 Hz source frequency. The sample temperatures are, respectively, 160, 200, 260 and 300 K for the black, red, green, and blue lines.

Application of the RRM method allowed us to simultaneously collect up to six inelastic spectra with different incoming wavelengths. Using WFM several incoming wavelength bands could be joined together for gap free coverage of about 9 Å bandwidth in TOF powder diffraction with chopper shaped pulses. The experimental data in both cases can be treated in conventional manner. Probing the sample with multiple wavelengths or multiple wavelength bands, however, can offer enhanced quality of information by broader dynamic range.

[1] F. Mezei, *J. Neutron Res*. 6 (**1997**) 3
[2] F. Mezei, M. Russina, *Advances in neutron scattering instrumentation*, in: I.S. Anderson, B. Guerard (Eds.), *Proceedings of SPIE*, vol. 4785, **2002**, p. 24.
[3] M.Russina, F. Mezei, *Nuclear Instr. and Methods*, A604 (**2009**) 624
[4] M. Russina, Gy. Kali, Zs. Santa, F. Mezei, *Nuclear Instr. and Methods* A, **2011**, doi:10.1016/j.nima.2011.05.077

Confinement effects on molecular hydrogen dynamics in ice-based clathrates

M.Russina, E.Kemner

Clathrate hydrates are inclusion compounds, formed by a network of hydrogen-bonded water molecules that is stabilized by the presence of foreign guest molecules. The hydrophobic interactions between the host framework and guest molecules push the ice to change its structure and form cages of different forms and dimensions to accommodate the guests. The topology of the ice cages depends on the sizes and shapes of the guest molecules, the typical dimensions of the cages range between 4 - 15 Å. The confinement and small size of the cages strongly influence the behavior of the guest molecules and results in properties different from those of the bulk, making clathrates hydrates model system to study the effects of confinement. There are indications that the size of the nanocage affects macroscopic properties important for gas storage, e.g. of hydrogen. For instance the loading pressure for the clathrates where hydrogen only occupies so-called small cages is reduced by almost one order of
magnitude and temperature stability increases by 100 K compared to the clathrates where hydrogen occupies cages of larger dimensions. The origin of such behavior lies however in the microscopic interactions between the hydrogen and the host.
To understand the reason of these differences we undertook a detailed study of the guest-host interactions by combining neutron scattering and molecular dynamics simulation. Guest-host interactions are best reflected in the microscopic dynamics, thus the benefits and complementarities of neutron scattering and molecular dynamics simulations are obvious: both techniques provide information on atomic trajectories allowing for a direct comparison of the theoretical and experimental results. Both methods work with full efficiency on the nanometer length scale relevant for hydrogen storage structures and can follow atomic motions on a broad time scale from 10^{-14} to 10^{-10} s.

We have investigated the behavior of molecular hydrogen in the bulk and confined into cages of different dimensions of ice-based clathrates. For this purpose we used the composite THF - H_2O - H_2 and H_2O - H_2 systems. THF stands for the tetrahydrofuran molecule. In the ternary clathrate the THF molecules take up the places in the larger cages, and the molecular hydrogen occupies only small cages of 5.02Å accessible diameter with one molecule / cage. In the fully hydrogenated H_2O - H_2 system hydrogen can occupy both the small and the large cages. The latter ones are of 6.67Å accessible diameter and accommodate two H_2 molecules per cage.

We have found that the confinement dimensions have strong impact on the mobility of the guest hydrogen molecules. On the one hand, we observed large differences between the values of the mean square displacements in the large and small cages, giving evidence for the localization of the guest molecules in the small cage [1]. On the other hand, in addition to the first rotational transition we observed a dynamic feature of the hydrogen in the small cage, consisting of several components in the 8-12 meV energy range [2], [3]. Remarkable is that such dynamic response of the molecular hydrogen is absent in the large cages.

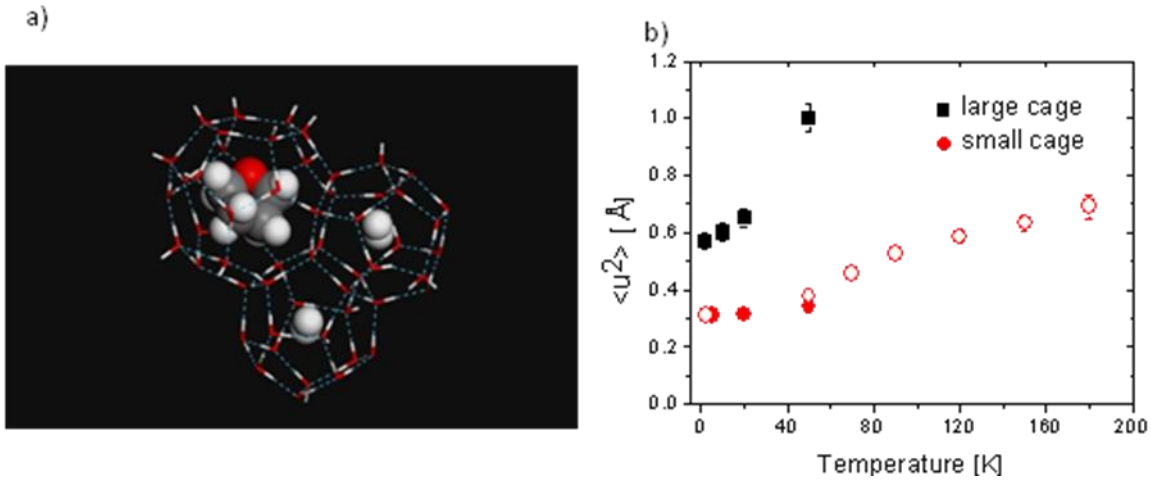


Fig.1 Structure of the THF-clathrate with small cages filled with one molecule of H_2 (a). In the fully hydrogenated clathrates two hydrogen molecules can occupy the large cages. (b) The mean-square displacement of the hydrogen molecule in the large and small cage as function of temperature.

The dynamic feature at 8-12 meV energy range has been also reported in several theoretical studies and was associated with translational, “rattling” modes. The assumption of contribution of translational modes is also supported by our results from molecular dynamic simulations. However, translational motion alone cannot explain the large intensity of this feature observed in the small cages. In fact the localization of the guest molecules in the small cage and the similarity between energies of rotational and translational motions leads to strong coupling between them and thus to the onset of new, hybrid type of motion. The strong coupling between modes and quantum localization of the hydrogen molecules expected to decrease the kinetic energy of the confined molecular hydrogen. In addition to observed localization it plays a role of the effective quantum “attachment” to the framework resulting in higher temperature-pressure stability of the entire H_2 clathrate system.

[1] M. Russina, E. Kemner et al, *in press*
[2] L. Ulivi, M. Celli, A. Giannasi, A. J. Ramirez-Cuesta, and M. Zoppi, *J.Phys.: Condens. Matter* 20, 104242 _**2008**
[3] M. Russina, E. Kemner, M. Cellie, L. Ulivi, F. Mezei, *J. Phys.: Conf. Ser*. 177 (**2009**) 012013

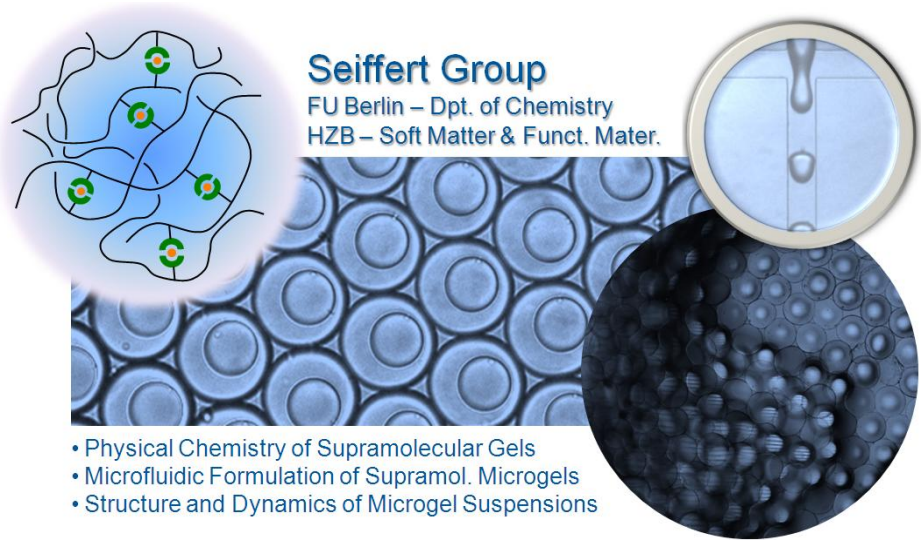
Polymer Physics

Dr. Sebastian Seiffert

Polymer gels consist of a three dimensional network of crosslinked polymer chains which are swollen in a solvent; they are fascinating materials for applications as superabsorbers, matrixes in analytical chemistry or biology, or as storage and delivery systems for actives. Classically, network junctions are formed by chemical bonds, ensuring great mechanical stability. However, these chemical crosslinks are permanent, such that chemical gels cannot be processed or recycled. Permanent chain interconnection is also detrimental for encapsulation and controlled release applications. It is therefore desirable to use *reversible* gels, which is readily achieved by *supramolecular* chain crosslinking. Previous work in this field has produced various types of such materials, primarily realized through chain interconnection by hydrogen bonding or transition metal complexation. However, a comprehensive characterization and deep understanding between the chemical structure and the phenomenological behavior of these promising materials is still lacking.

Our research focuses on polymer networks that are crosslinked by supramolecular bonds. We prepare, study, and process these networks in a systematic fashion. For this purpose, we use a universal covalent precursor polymer and equip it with side groups that can be interconnected by non-covalent interactions such as hydrogen bonding or transition metal complexation. This leads to networks that consist of the same basis material, yet exhibiting a strongly varying strength of chain interconnection.

We study these supramolecular gels to derive fundamental relations between the strength of non-covalent crosslinking and the network structure and dynamics, using methods such as macroscopic rheology as well as light, neutron, and x-ray scattering. In addition, we use heterophase techniques such as miniemulsification and droplet-based microfluidics to fabricate supramolecular nano- and microgel particles. These particles can serve as nano- and microcapsules for the encapsulation and controlled release of actives, including drugs, biopolymers, and living cells. We also use these particles as microscopic probes to study their polymer network architectures, and we investigate the physical chemical properties of densely packed suspensions of these micro- and nanogels.



Coworkers:

Dr. Sebastian Seiffert

Sebastian Hackelbusch

Fany Di Lorenzo

M.Sc. Torsten Rossow

Hendrik Ronneburg

Publications in 2010–2012:

T. Liu, S. Seiffert, J. Thiele, A. R. Abate, D. A. Weitz, W. Richtering, “Non-Coalescence of Oppositely Charged Droplets in pH-Sensitive Emulsions.” *Proc. Natl. Acad. Sci. U.S.A.* **2012**,*109*, 384–389.

P. Menut, S. Seiffert, J. Sprakel, D. A. Weitz, “Does Size Matter? Elasticity of Compressed Suspensions of Colloidal- and Granular-Scale Microgels.” *Soft Matter* **2012**, *8*, 156–164.

S. Seiffert and J. Sprakel, “Physical chemistry of supramolecular polymer networks.” *Chem. Soc. Rev.* **2012**, *41*, 309–330.

S. Seiffert, “Functional Microgels Tailored by Droplet-Based Microfluidics.” *Macromol. Rapid. Commun.* **2011**, *32*, 1600–1609.

J. Thiele and S. Seiffert, "Double emulsions with controlled morphology by microgel scaffolding." *Lab Chip* **2011**, *11*, 3188–3192.

A. R. Abate, M. Kutsovsky, S. Seiffert, M. Windbergs, L. F. V. Pinto, A. Rotem, A. S. Utada, D. A. Weitz, "Synthesis of Monodisperse Microparticles from Non-Newtonian Polymer Solutions with Microfluidic Devices." *Adv. Mater.* **2011**, *23*, 1757–1760.

S. Seiffert, J. Dubbert, W. Richtering, D. A. Weitz, "Reduced UV light scattering in PDMS microfluidic devices." *Lab Chip* **2011**, *11*, 966–968.

D. Steinhilber, S. Seiffert, J. A. Heyman, F. Paulus, D. A. Weitz, R. Haag, “Hyperbranched polyglycerols on the nanometer and micrometer scale.” *Biomaterials* **2011**, *32*, 1311–1316.

S. Seiffert, D. A. Weitz, “Microfluidic fabrication of smart microgels from macromolecular precursors.” *Polymer* **2010**, 51, 5883–5889.

S. Seiffert, M. B. Romanowsky, and D. A. Weitz, “Janus Microgels Produced from Functional Precursor Polymers.” *Langmuir* **2010**, *26*, 14842–14847.

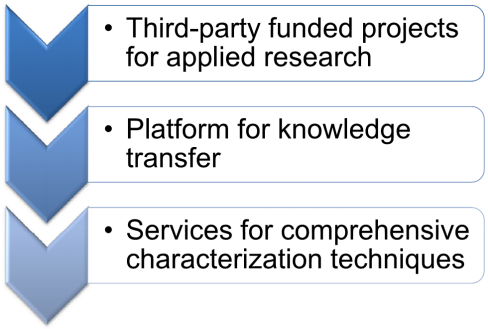
S. Seiffert and D. A. Weitz, “Controlled Fabrication of Polymer Microgels by Polymer-Analogous Gelation in Droplet Microfluidics.” *Soft Matter* **2010**, *6*, 3184–3190.

S. Seiffert, J. Thiele, A. R. Abate, and D. A. Weitz, “Smart Microgel Capsules from Macromolecular Precursors.” *J. Am. Chem. Soc.* **2010**, *132*, 6606–6609.

Industry Relations

Martin Hoffmann

Industry relations acts as an interlock between particular research activities at the institute F-I2 and external partners with a strong interest in industrial relevant issues. Within the Institute of Soft Matter and Functional Materials (F-I2), industry relations is closely collaborating with the Colloid Chemistry and the Colloid Physics group. We provide expertise in the field of the synthesis and comprehensive characterization of polymer (hybrid) colloids to external cooperation partners from industry. We are therefore open to:



Within the third-party funded applied research, industry relations is participating in two projects at the moment. The first one is the large scale integrating collaborative project POLYCAT within **FP 7** of the EU. Here, Dr. Martin Hoffmann is looking at the task how to use polymer-based catalysts under microflow conditions in order to perform fine chemical synthesis. The second project is focused on the development and commercialization of separation technologies for ultra-small nanoparticles by asymmetric flow field flow fractionation (Dr. Alexei Plotnikov). For both projects, an overview will be given later.

Within industry relations, a platform for knowledge transfer will be established on the homepage of the institute F-I2. One major issue is the organization of the advanced training course “The Chemistry and Physics of Polymer Dispersions” on the HZB campus Wannsee in 2012/2013 in cooperation with the “Deutsche Gesellschaft für Materialkunde e. V.” (**DGM**).

The advanced characterization of nanosized products from an industrial relevant process often demands deep knowledge in the characterization technique as well as expensive instruments. Within F-I2, industry relations can provide comprehensive analytical techniques to external partners within the frame of (long term) cooperations. In addition to neutron and X-ray scattering, analytical techniques include (dynamic) light scattering (DLS) with the option to measure the depolarized light component. The measurement of the electrophoretic mobility / ζ-potential is an indispensable tool for quantifying the colloidal stability in solution (Malvern Zetasizer Nano-ZS). Inductively coupled plasma mass spectroscopy (ICP-MS) can be provided as well.

Coworkers

Dr. Martin Hoffmann
Dr. Alexei Plotnikov
M.Sc. Bin Dai

Selected Publications in 2009-2011

[1] Lu, Y.; Hoffmann, M.; Yelamanchili, R. S.; Terrenoire, A.; Schrinner, M.; Drechsler, M.; Möller, M. W.; Breu, J; Ballauff, M. Well-defined crystalline TiO2-Nanoparticles Generated and Immobilized on a Colloidal Nanoreactor. *Macromol. Chem. Phys.* **2009**, *210*, 377.

[2] Lesnyak, V.; Lutich, A.; Gaponik, N.; Grabolle, M.; Plotnikov, A.; Resch-Genger, U.; Eychmuller, A. *J.Mater. Chem.* **2009**, *19*, 9147.

[3] Hoffmann, M.; Siebenbürger, M.; Harnau, L.; Hund, M.; Hanske, C.; Lu, Y.; Wagner, S.; Drechsler, M.; Ballauff, M. Thermoresponsive Colloidal Molecules. *Soft Matter*, **2010**, *6*, 1125.

[4] Lesnyak, V.; Dubavik, A.; Plotnikov, A.; Gaponik, N.; Eychmuller, A. *Chem.Commun.* **2010**, *46*, 886.

[5] Jiménez, M. L.; Delgado, A. V.; Ahualli, S.; Hoffmann, M.; Wittemann, A.; Ballauff, M. Giant permittivity and dynamic mobility observed for spherical polyelectrolyte brushes. *Soft Matter* **2011**, *7*, 3758.

[6] Schürer, B.; Hoffmann, M.; Wunderlich, S.; Harnau, L; Peschel, U.; Ballauff, M. and Peukert, W. Second Harmonic Light Scattering from Spherical Polyelectrolyte Brushes. *J. Phys. Chem. C*. **2011**, *115*, 18302.

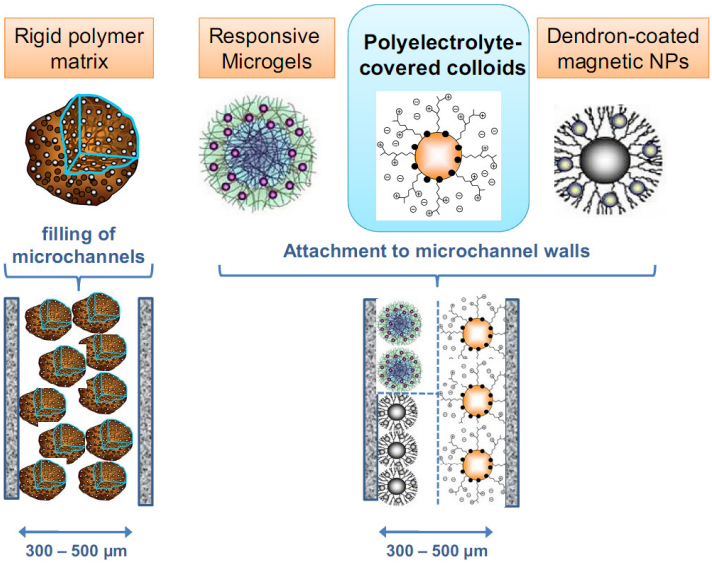
POLYCAT: Modern polymer-based catalysts and microflow conditions as key elements of innovations in fine chemical syntheses*

Martin Hoffmann

POLYCAT is an EU-funded FP7 grant to apply novel polymer based nanoparticle catalysts for the synthesis of chemicals in the pharmaceutical industry and crop protection by making use of micro process technology under environmentally benign conditions. The large-scale integrated project started in 2010-10-01 with a duration of 42 months, costs of 10.06 million Euro and a project funding of 7.00 million Euro where the EU contribution for the institute F-I2 at HZB amounts to 342,919.00 Euro.[1]

The interdisciplinary project covers fine chemistry, catalysis and engineering and is managed and operated by an international consortium. Nanosized catalysts are expected to simplify chemical or microbiological processes due to their advanced activity, selectivity, stability and recyclability. Micro process technology in comparison to batch-conditions offers the advantage of improved heat transfer with the option for scale-out. Three processes shall demonstrate the industrial applicability by scale-out to the pilot scale. As released on the official project homepage, “a multi-purpose, container-type plant infrastructure will integrate individual reaction and separation modules in block format, standardized basic logistics, process control, safety installations, and on-line analytics. As guidance during the whole development, holistic life cycle (LCA) and cost analyses will pave directions towards competitiveness and sustainability.”[1]

Within POLYCAT, Dr. Martin Hoffmann is a work package leader and responsible for the synthesis, characterization and application of organic or hybrid catalytically active polyelectrolyte covered colloids. These shall be immobilized in microchannels with different surface chemistry (see Figure) and used in the synthesis of industrially relevant fine-chemicals in cooperation with Sanofi-Aventis Recherche & Developpement (compare [2]).



[1] http://www.polycat-fp7.eu/ (Date: 2012-02-15)
[2] Proch, S.; Mei, Y.; Rivera Villanueva, J. M.; Lu, Y.; Karpov, A.; Ballauff M.; Kempe, R. *Adv. Synth. Catal*. **2008**, *350*, 493.

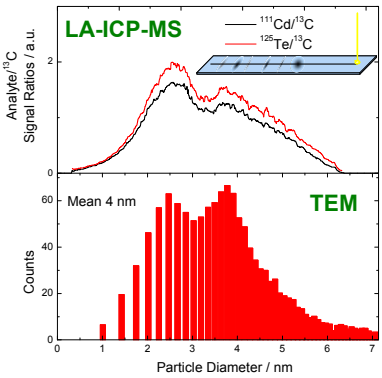
* The research leading to these results has received funding from the European Community's Seventh Framework Programme [FP7/2007-2013] under grant agreement no. CP-IP 24095

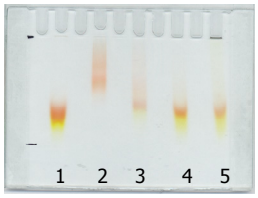
Complex characterization of nanocolloids by hyphenation of various separation techniques with inductively coupled plasma mass spectrometry

Dr. Alexei Plotnikov, Dr. Vladimir Lesnyak*, Dr. Nikolai Gaponik*

High photoluminescence quantum efficiency of nanosized semiconductors makes them very attractive materials for modern photonics or biological labeling for fluorescent imaging.

Electronic properties of nanocrystals are size-dependent and can drastically change within a narrow size range (approx. 1-10 nm). The investigation of size-dependent properties as well as the optimization of the synthesis procedures of colloids containing stabilized semiconductor nanocrystals requires the information both on the size distribution of constituent particles and their elemental composition.[1,2] However the techniques recently used for these purposes do not satisfy all the requirements being either time-consuming or inaccurate, or insufficiently informative or not well suited for the characterization of particles smaller than 10 nm. Thus the problem of express simultaneous analysis of both the morphology and elemental composition still remains a challenge.





1. 2.5 nm CdTe
2. 4 nm CdTe
3. mixture of 1) and 2)
4. 3 nm CdTe + 3.5 nm CdSeTe
5. 3 nm CdTe + 3.5 nm CdSeTe + 7nm ZnCdTe

A versatile approach to the rapid characterization of nanocolloids involving electrophoresis in gels with consequent scanning by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) was developed in the cooperation between HZB and TU Dresden. Different nanocolloids with the mean particle diameter between 1 and 10 nm were separated electrophoretically in polyacrylamide gels with cross-linkage degree of 8% to evaluate the practical applicability of the suggested approach. The lanes containing nanoparticle fractions were cut after gel drying, fixed on a glass substrate and scanned with a constant velocity along the migration line by a pulsed UV-laser in a closed He-flushed chamber connected to the ICP-MS. The spatially-resolved profiles of the main constituents of the CdTe colloid are in a good agreement with the data obtained by electron microscopy (Figure). The approach allows a simultaneous determination of the particle size distribution and the size-dependent variation of particle composition, which is not possible only by means of optical methods.

Additionally the theoretical basics involving a deconvolution procedure for the retrieval of the particle size distribution from the measured transient data was developed.

Further developments include the application of a combined optimized separation channel for asymmetrical flow / electrical field-flow fractionation connected to the ICP-MS. The use of a switchable separation principle should allow obtaining the particle size distribution as well as the size-related data on chemical composition and electrokinetic properties for the investigated nanocolloids.

[1] Lesnyak, V.; Lutich, A.; Gaponik, N.; Grabolle, M.; Plotnikov, A.; Resch-Genger, U.; Eychmuller, A. *J.Mater. Chem.* **2009**, *19*, 9147.

[2] Lesnyak, V.; Dubavik, A.; Plotnikov, A.; Gaponik, N.; Eychmuller, A. *Chem.Commun.* **2010**, *46*, 886.

* Technische Universität Dresden, Department of Physical Chemistry / Electrochemistry

Soft Matter and Functional Materials

CVs

Prof. Dr. Matthias Ballauff

(1952)

F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin
and
Humboldt-Universität zu Berlin,
Institute of Physics, Newtonstrasse 15, 12489 Berlin
Tel: 030 8062-43071 (-42308, Fax)
Email: matthias.ballauff@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise:	Physics of colloids and macromolecules
Field of Interest:	Chemistry and physics of colloidal systems, scattering methods

Scientific Development

Study	1971- 1977 Study of chemistry at the University of Mainz
PhD	1981 at the University of Mainz (Prof. Dr. B. A. Wolf)
Post-doc	1981-1983 Dept. Chemistry, Stanford University, Prof. Dr. P. J. Flory
	1984- 1989 Research Associate, Max-Planck-Institut für Polymerforschung, Mainz
	1989 Habilitation at University of Mainz
Full Chair	1990- 2003 at University of Karlsruhe
	2003- 2009 at University of Bayreuth
	Since 2009 Head of the Institute for Soft Matter and Functional Materials, HZB, Berlin, and Professor of Physics, Humboldt Universität zu Berlin

Selected Scientific Expert, Review or Council Activities, Awards

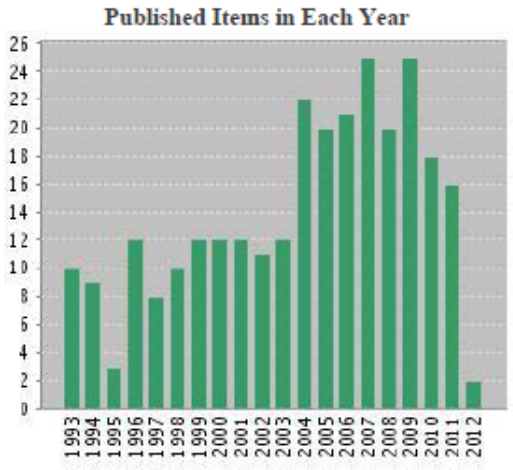
Member of the DFG-Panel for the special research areas of the Deutsche Forschungsgemeinschaft (Mitglied des DFG Ausschusses für die Angelegenheiten der Sonderforschungsbereiche) (1999-2005), Themenkommission Deutsche Bunsen-Gesellschaft (2008 – 2011), Member of „Ständiger Ausschuss“ Deutsche Bunsen-Gesellschaft (2011 – 2012), Editor Polymer (since 2004), Scientific Advisory Committee HERCULES, Fachkollegiat DFG since 2012

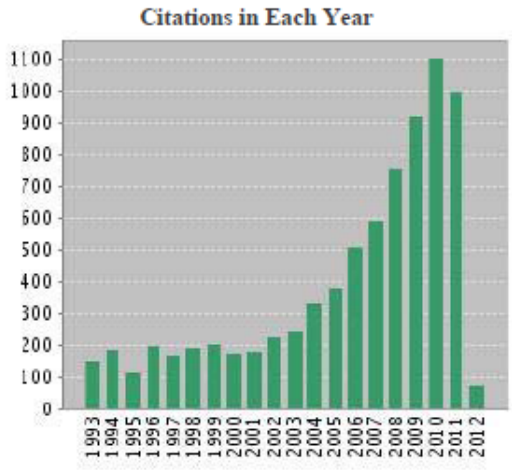
Invited Talks (selection)

“43rd IUPAC World Polymer Congress Macro”, Glasgow, England 2010; “23 General Conference of the European Physical Society”, Warschau, Polen 2010; “RUSNANOTECH Forum”, Moskau, Russland 2010; “8th International Symposium on Polyelectrolytes”, Shanghai, China 2010, Smart Polymer Systems Conference, Mainz 2011, 7th International Symposium “Molecular Mobility and Order in Polymer Systems”, St. Petersburg, Russland 2011

Selected publications

1. N. Welsch, A. L. Becker, J. Dzubiella, M. Ballauff, *Core-shell microgels as „smart" carriers for enzymes*, Soft Matter 2012, 8, 1428,1436
2. A. Zaccone, J. J. Crassous, B. Beri, M. Ballauff, *Quantifying the Reversible Association of Thermosensitive Nanoparticles*, Phys. Rev. Lett. 2011, 107, 168303
3. S. Wunder, Y. Lu, M. Albrecht, M. Ballauff, *Catalytic Activity of Faceted Gold Nanoparticles Studied by a Model Reaction: Evidence for Substrate-Induced Surface Restructuring,* ACS Catalysis 2011, **1**, 908.
4. J. M. Brader, M. Siebenbürger, M. Ballauff, K. Reinheimer, M. Wilhelm, S. J. Frey, F. Weysser, M. Fuchs, *Nonlinear response of dense colloidal suspensions under oscillatory shear: Mode-coupling theory and Fourier transform rheology experiments,* Phys. Rev. E 2010, **82**, 061401
5. M. Hoffmann, M. Siebenbürger, L. Harnau, M. Hund, C. Hanske, Y. Lu, C. S. Wagner, M. Drechsler,M. Ballauff, *Thermoresponsive colloidal molecules*, Soft Matter, 2010, **6**, 1125-1128
6. K. Henzler, B. Haupt, K. Lauterbach, A. Wittemann, O. Borisov,M. Ballauff, *Adsorption of beta-Lactoglobulin on Spherical Polyelectrolyte Brushes: Direct Proof of Counterion Release by Isothermal Titration Calorimetry*, J Am Chem Soc, 2010, **132**, 3159-3163
7. M. Schrinner, M. Ballauff, Y. Talmon, Y. Kauffmann, J. Thun, M. Möller, J. Breu, *Single-Nanocrystals of Platinum Prepared by Partial Dissolution of Au-Pt Nanoalloys,* Science, **323**, (2009) 617.
8. K. Henzler, S. Rosenfeldt, A. Wittemann, L. Harnau, S. Finet, T. Narayanan, M. Ballauff, *Directed motion of proteins along tethered polyelectrolytes,* Phys. Rev. Lett., **100**, (2008) 158301.
9. M. Ballauff, *Spherical Polyelectrolyte Brushes*, Progr. Polym. Sci., **32**, (2007) 1135.
10. Y. Lu, Y. Mei, M. Drechsler, M. Ballauff, *Thermosensitive Core-Shell Particles for Ag-Nanoparticles: Modulating the Catalytic Activity by the Volume Transition in Networks,* Angew. Chemie Intl. Ed. **45**, (2006) 813.





Results found:	**328**
Sum of the Times Cited:	**8156**
Average Citations per Item:	**24.87**
h-index:	**45**

Dr. Daniel Clemens

(1961)

F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin
Tel: 030 8062-42280 (-42651, Fax)
Email: clemens@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise:	Neutron and X-ray instrumentation
Fields of Interest:	Physics of colloidal systems, Deposition and characterization of multilayer systems

Scientific Development

Study	1981- 1989 Study of physics at the Technische Universität Berlin
PhD	1993 at the Technische Universität Berlin (Prof. Dr. F. Mezei)
Post-doc	1994-2001 Laboratory for Neutron Scattering Villigen, Paul Scherrer Institut and ETH Zürich, Prof. Dr. A. Furrer / Prof. Dr. P. Böni
Staff positions	2001- 2002 Staff scientist, Laboratory for Neutron Scattering Villigen, Paul Scherrer Institut and ETH Zürich
	Since 2002 Staff scientist, Dept. Methods and Instrumentation for Neutron Scattering and Institute for Soft Matter and Functional Materials, HZ Berlin

Selected Scientific Expert, Review or Council Activities, Awards

International advisory panel of Int. Small-angle Scatt. Conf. SAS-2012, Sydney (2010-2012), International advisory panel of the Laboratoire Léon Brillouin of CEA/CNRS, Saclay (since 2010), Scientific & techn. advisory panel for SANS instruments at the European Spallation Source (ESS) (since 12.2011), Elected member of Scientific & technology board of HZB (02-12.2012)

Selected publications

[1] V. Mengarelli, M. Zeghal, L. Auvray, D. Clemens: *Phase behavior and structure of stable complexes between a long polyanion and a branched polycation*, Physical Review E **84**, 1-12 (2011)

[2] Y. Gerelli, M.T. Di Bari, A. Deriu, D. Clemens, L. Almásy, *Lipid multilayered particles: the role of chitosan on structure and morphology*, Soft Matter **6**, 2533–2538 (2010)

[3] F. Cousin, J. Gummel, D. Clemens, I. Grillo, F. Boué, *Multiple Scale Reorganization of Electrostatic Complexes of Poly(styrenesulfonate) and Lysozyme*, Langmuir, **26**, 7078–7085 (2010)

[4] M.G. Ortore, R. Sinibaldi, F. Spinozzi, F. Carsughi, D. Clemens, A. Bonincontro, P. Mariani, *New Insights into Urea Action on Proteins: A SANS Study of the Lysozyme Case,* J. Phys. Chem. B **112**, 12881-12887 (2008).

[5] I. Estrela-Lopis, S. Leporatti, E. Typlt, D. Clemens, E. Donath, *Small Angle Neutron Scattering Investigations (SANS) of Polyelectrolyte Multilayer Capsules Templated on Human Red Blood Cells*, Langmuir, **23**, 7209–7215 (2007).

Prof. Dr. Joachim Dzubiella

(1975)

F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin
and
Humboldt-Universität zu Berlin,
Institute of Physics, Newtonstrasse 15, 12489 Berlin
Tel: 030 8062-42902 (-42308, Fax)
Email: joachim.dzubiella@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise: Theoretical Physics of Complex Fluids

Field of Interest: Structure and dynamics of colloids, polymers, and proteins

Scientific Development

1994-1999	Study of Physics at the HH-University of Düsseldorf
2002	PhD at the HH-University of Düsseldorf (Prof. Dr. H. Löwen)
2002-2004	Postdoc Dept. Chemistry, Cambridge University, UK, Prof. Dr. J.-P. Hansen
2004-2006	Postdoc Dept. Biochemistry, Fellow of the Center for Theoretical Biophysics, UC San Diego, USA (Prof. J. A. McCammon).
2006-2010	Emmy-Noether Fellow, Research Group Leader, Physics Dept., Technical University Munich
Since 2010	Group Leader at the Soft Matter and Functional Materials Institute, HZB
Since 2011	Professor of Theoretical Physics, Humboldt Universität zu Berlin

Scholarships

2002 DAAD Auslandsstipendium (UC Santa Barbara)

2004 DFG Forschungsstipendium (UC San Diego)

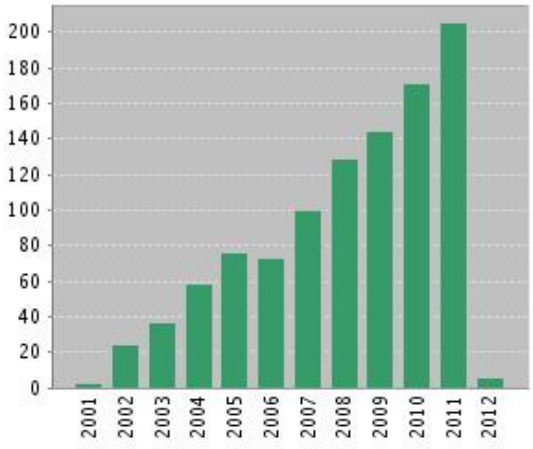
2006 DFG Emmy-Noether Fellowship (TU Munich)

Invited Talks (selection)

2007, UTAM Symposium on Swelling and Shrinking of Porous Materials, Rio de Janeiro, Brazil; 2008, CTBP summer school “Coarse-Grained Physical Modeling of Biological Systems, UCSD, USA; 2009, International Workshop on Continuum Modeling of Biomolecules, Beijing, China; 2010 Gordon Research Conference on “Aqueous Systems”, USA; 2010 Hydrophobicity session at Pacificchem, Honolulu, USA; 2011, Telluride workshop on osmolytes, USA; 2012 ACS San Diego;

Selected publications

1) Effects of Hofmeister ions on the α-helical structure of proteins
   A. H. Crevenna, N. Naredi-Rainer, D. C. Lamb, R. Wedlich-Söldner, J. Dzubiella, *Biophys. J.,* to be published (2012).
2) 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, *Angewandte Chemie*, to be published (2012).
3) Ionic-specific excluded-volume correlations and solvation forces
   I. Kalcher, J. C. F. Schulz, and J. Dzubiella, *Phys. Rev. Lett.* **104**, 097802 (2010).
4) Salt-specific stability and denaturation of a short salt-bridge forming alpha-helix
   J. Dzubiella, *J. Am. Chem. Soc.* **130**(42), 14000-14007 (2008).
5) Nonequilibrium sedimentation of colloids on the particle scale, . Royall, J. Dzubiella, M. Schmidt, and A. van Blaaderen, *Phys. Rev. Lett.* **98**, 188304 (2007).



Citations per year as of February, 2012. Total citations ~1000.
H-index = 20.

Dr. Günter Johannes Goerigk

(1953)

F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, D-14109
Berlin
Tel: 030 8062-15149 (-15752, Fax)
Email: guenter.goerigk@helmholtz-berlin.de

Area of Expertise and Fields of Interest

Area of Expertise: Anomalous Small-Angle X-ray Scattering, Very Small-Angle Neutron Scattering, Decomposition kinetics in alloys

Fields of Interest: Time resolved in-situ ASAXS studies on the decomposition kinetics of Copper-Cobalt alloys. Extensive investigations of amorphous Silicon-Germanium alloys used in solar cell techniques. Analysis of conformational and quantitative properties of counter ion condensation to polyelectrolytes by anomalous small-angle X-ray scattering. Critical scattering of amorphous ternary metallic alloys. Critical Casimir effect in binary liquid mixtures analyzed by V-SANS (Very Small-Angle Neutron Scattering). Numerous ASAXS studies on alloys, ceramics, magnetic systems, catalysts, semiconductors, glasses, polymers, membranes and soft matter systems in collaboration with scientific groups from Europe, United States, South America and Asia.

Scientific Development

Study	1973 - 1982	Study of physics, Dipl.-Phys. at University of Bonn
PhD	1988	in chemistry at University of Hamburg (Prof. Dr. H. G. Zachmann, Prof. Dr. H.B. Stuhrmann)
Post-doc	1988 -1990	GKKS Research Centre, Institute of Materials Research
Synchrotron radiation	1983 -1984	ASAXS beam line X15 at EMBL Outstation at DESY Hamburg, Prof. Dr. H.B. Stuhrmann
	1985 - 1990	Establishing ASAXS beam line ROEFO1 together with B. Munk at HASYLAB/DESY Hamburg
	1991 - 2007	Scientist in charge at ASAXS beam line JUSIFA @HASYLAB/DESY Hamburg, Forschungszentrum Jülich
Neutrons	since 2007	Scientist at V-SANS beam line KWS-3@JCNS/FRM II Munich, Forschungszentrum Jülich, Comprehensive upgrade activities, commissioning, implementation into the regular FRM II proposal and scheduling system since December 2009
	Oct. 2010	Member of F-I2 Institute of Soft Matter and Functional Materials, Helmholtz-Zentrum Berlin

Selected Scientific Expert, Review or Council Activities

1996-2005	Member of the scientific committee of DESY
2003-2007	Project leader JUSIFA@HASYLAB
2007-2010	Member of the co-ordination panel JCNS instrumentation at FRM II
Since 2011	Peer Review Panel member of Diamond Light Source ASAXS-consultant

Invited Talks (selection)

Goerigk, G., Anomalous Small-Angle X-ray Scattering: A Precise Quantitative Method in Chemistry and Solid State Physics, Hungarian Academy of Science, MTA Kemial Kutatoközpont, Budapest, Hungary, 30.06.2009

Goerigk, G., Anomalous Small-Angle X-ray Scattering: A Precise Quantitative Method in Condensed Matter Research, Invited talk to be held at REXS 2011, International Conference/School on Resonant Elastic X-Ray Scattering in Condensed Matter, Aussois, France; 13.-17. June 2011

Goerigk, G., Small-Angle X-ray Scattering with Synchrotron Radiation and Neutrons – Precise Experimental Techniques for Quantitative and Structural Analysis in Chemistry and Physics, Invited talk to be held at TMS 2012, 141st Annual Meeting, Orlando Florida, U.S.A.; 11.-15. March 2012

Selected publications

Bota, A.; Varga, Z.; Goerigk, G..: Biological systems as nanoreactors: Anomalous small-angle X-ray scattering study of the CdS nanoparticle formation in multi-lamellar vesicles. Journal of Physical Chemistry B (2007), 111(8), 1911-1915.

Goerigk, G.; Huber, K.; Schweins, R. (2007): Probing the extent of the Sr2+ ion condensation to anionic polyacrylate coils: A quantitative anomalous small-angle x-ray scattering study. Journal of Chemical Physics, 127(15), 154908/1-154908/8.

Goerigk, G.; Mattern, N. Critical scattering of Ni-Nb-Y metallic glasses probed by quantitative anomalous small-angle x-ray scattering. Acta Materialia (2009), 57(12), 3652-3661.

Goerigk, G., Varga, Z., Comprehensive upgrade of the high-resolution small-angle neutron scattering instrument KWS-3 at FRM II, Journal of Applied Crystallography (2011), 44, 337-342

Lectures at University Paderborn, Department of Chemistry

In summer semester 2010 in Physical Chemistry contributing with 8 hours to the lecture ‘Structure determination’ of Prof. K. Huber.

In addition to the lecture ‘Structure determination’ of summer semester 2010 practical exercises with 4 students of Universität Paderborn at Jülich Centre of Neutron Science (JCNS) at the Research reactor FRM II@TUM in Garching/Munich. at V-SANS beam line KWS-3. Three days exercises, seminar and tutorials.

In summer semester 2011 lectures and exercises ‘Structure determination’ at University Paderborn, Lehrauftrag for 13 weeks, 2 hours per week.

In addition to the lecture ‘Structure determination’ of summer semester 2011 practical exercises with 9 students of Universität Paderborn at Jülich Centre of Neutron Science (JCNS) at the Research reactor FRM II@TUM in Garching at V-SANS beamline KWS-3. Three days exercises, seminar and tutorials.

Scientific(selected) co-operations

Prof. K. Huber, Universität Paderborn, Fakultät für Naturwissenschaften, Department Chemie, Warburgerstr. 100, D-33098 Paderborn, Federal Republic of Germany, Combined SANS, ASAXS and LS Studies of Structural Transformation in Polyacrylate Anions Induced by Specifically Interacting Metal Cations.

Dr. A. Bóta, Department of Biological Nanochemistry, Chemical Research Center, Hungarian Academy of Sciences, Pusztaszeri ut 59-67, H-1025, Budapest, Hungary, Structure and interparticle interactions in biomedical colloidal systems

Dr. Nikoline Hansen

(1958)

F-I2 Soft Matter and Functional Materials
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin

Tel: 030 8062-43074 (-42308, Fax)
Email: nikoline.hansen@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise:	Print and New Media, Assistance
Field of Interest:	Organisation and Consulting

Scientific Development

5/1986	Magister in American Studies, Politics as well as Prehistoric and Protohistoric Archaeology
1/1996	PhD at the Freie Universität Berlin, American Studies Public Relations, DAPR New Media, Fachhochschule für Technik und Wirtschaft
06/1986-05/2009	Team Secretary at BESSY and BESSY II Project, Personal Assistant to Professor Jaeschke (Director, accelerator physics)
09/1997-101998	Night School Public Relations, DAPR Examination PR Consultant
11/2001-03/2003	Advanced off-the-job training in Visual Computing and Web Technology (Medieninformatik) at the Fachhochschule für Technik und Wirtschaft Berlin
Since 06/2009	Institute Soft Matter and Functional Materials, administrative officer, Helmholtz-Zentrum Berlin, Germany, Prof. Dr. M. Ballauff

Print Media:
BESSY II – Eine optimierte Undulator/Wiggler-Speicherring Lichtquelle für den VUV- und XUV-Spektralbereich (1986)
Visions of Science: the BESSY SASE-FEL in Berlin-Adlershof Scientific Case for an FEL (2001)
Technical Design Report BESSY FEL (2004), BESSY, Berlin

International Conference Organisation
FEL06 (Local Organisation Committee and Conference Secretary Berlin 2006)

Organisation of International Committees and Workshops
BESSY II MAC (1993-1998, 11 Meetings)
Workshop on Scientific Case for the BESSY SASE-FEL, Holzhau 2001

Dr. Thomas Hauß

(1958)

F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin

Tel: 030 8062-42071 (-42999, Fax)
Email: hauss@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise:	Biophysics
Field of Interest:	Membrane biophysics, peptide-membrane interactions, protein dynamics, neutron scattering methods

Scientific Development

Study	1977- 1986 Study of physics, Technische Universität Berlin
PhD	1992 Freie Universität Berlin (Prof. Dr. M.P. Heyn)
Post-doc	1993-1996 Hahn-Meitner-Institut Berlin, Prof. Dr. M. Steiner
	1996-1999 Research Scientist, Clemens-Schöpf-Institute, Technische Universität Darmstadt, Prof. Dr. N.A. Dencher
	2000-2004 Research Scientist, Dept. Physics, Heinrich-Heine Universität Düsseldorf, Prof. Dr. G. Büldt
	2005-2009 Research Scientist, Clemens-Schöpf-Institute, Technische Universität Darmstadt, Prof. Dr. N.A. Dencher
permanent	Since 2009 Group leader Biophysics, Institute Soft Matter and Functional Materials, Helmholtz-Zentrum Berlin für Materialien und Energie

Selected Scientific Expert, Review or Council Activities, Awards

Reviewer for: Biotechnology and Biological Sciences Research Council (BBSRC), UK; NIST Center for Neutron Research, Gaithersburg, USA; DLab, ILL Grenoble, France.

Invited Talks (selection)

Workshop on (Glyco)lipids, structures, functions, and interactions, Universität Hamburg, Germany 2010; Neutrons in Biology, Lund University Sweden 2009; 13th International Conference on Retinal Proteins, Barcelona, Spain 2008; 3rd Japanese-French Seminar on Protein Dynamics, Grenoble, France 2007; 12th International Conference on Retinal Proteins - Satellite Meeting Nagoya, Japan 2006

Selected publications

[1] A. Buchsteiner, T. Hauß, S. Dante, N. A. Dencher. *Alzheimer's disease amyloid-beta peptide analogue alters the ps-dynamics of phospholipid membranes.* Biochimica et Biophysica Acta **1798** (2010), 1969-1976

[2] A. Schröter, D. Kessner, M. A. Kiselev, T. Hauß, S. Dante, R. H. H. Neubert. *Basic nanostructure of stratum corneum lipid matrices based on ceramides [EOS] and [AP]. A neutron diffraction study*. Biophysical Journal **97** (2009) 1104-14.

[3] H. Seelert, D. N. Dani, S. Dante, T. Hauß, F. Krause, E. Schafer, M. Frenzel, A. Poetsch, S. Rexroth, H. J. Schwaßmann, T. Suhai, J. Vonck, N. A. Dencher. *From protons to OXPHOS supercomplexes and Alzheimer's disease: Structure-dynamics-function relationships of energy-transducing membranes.* Biochimica et Biophysica Acta **1787**, (2009) 657-671.

[4] J. Pieper, A. Buchsteiner, N. A. Dencher, R. E. Lechner, T. Hauß. *Transient protein softening during the working cycle of a molecular machine.* Physical Review Letters **100**, (2008) 228103

[5] S. Dante, T. Hauß, A. Brand, N. A. Dencher. *Membrane fusogenic activity of the Alzheimer's peptide Aβ(1–42) demonstrated by small-angle neutron scattering.* Journal of Molecular Biology **376**, (2008) 393-404.

[6] S. Dante, T. Hauß, N. A. Dencher. 2006. *Cholesterol inhibits the insertion of the Alzheimer's peptide Aβ(25-35) in lipid bilayers*. European Biophysics Journal **35**, (2006) 523-531

Dr. Katja Henzler
(1980)

F-I2 Soft Matter and Functional Materials
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin

Tel: 030 8062-43039 (-42308, Fax)
Email: katja.henzler@helmholtz-berlin.de

Area of Expertise

protein adsorption onto colloids, colloidal chemistry and physics, small angle scattering techniques, isothermal titration calorimetry

Scientific Development

10/1998 – 07/2002	Study of Applied Chemistry with Marketing at the Fachhochschule Reutlingen
10/2003 – 07/2006	Study of Polymer- and Colloid Chemistry at the University of Bayreuth
09/2006 – 06/2010	PhD at the University of Bayreuth and the Helmholtz Centre Berlin (Prof. Dr. M. Ballauff): "Interaction of Proteins with Spherical Polyelectrolyte Brushes"
Since 07/2010	Postdoc at the Institute of Soft Matter and Functional Materials, in the X-ray microscopy group

Selected Publications

- Wagner, C.S.; Shehata, S.; **Henzler, K.;** Yuan, J.Y.; Wittemann, A.: Towards nanoscale composite particles of dual complexity. *J. Colloid Interface Sci*., **2011**, *355*, 115
- **Henzler, K.;** Haupt, B.; Lauterbach, K.; Wittemann, A.; Borisov, O.; Ballauff, M.: Adsorption of beta-Lactoglobulin on Spherical Polyelectrolyte Brushes: Direct Proof of Counterion Release by Isothermal Titration Calorimetry. J. Am. Chem. Soc., **2010**, *132*, 3159.
- **Henzler, K.;** Haupt, B.; Ballauff, M.: Enzymatic activity of immobilized enzyme determined by isothermal titration calorimetry. *Anal. Biochem.*, **2008**, *378,184*.
- **Henzler, K.;** Rosenfeldt, S.; Wittemann, A.; Harnau, L.; Finet, S.; Narayanan, T.; Ballauff, M.: Directed motion of proteins along tethered polyelectrolytes. Phys. Rev. Lett., **2008**, *100*, 158301.
- **Henzler, K.;** Wittemann, A.; Breininger, E.; Ballauff, M.; Rosenfeldt, S.: Adsorption of bovine hemoglobin onto spherical polyelectrolyte brushes monitored by small-angle x-ray scattering and Fourier transform infrared Spectroscopy. *Biomacromolecules*., **2007**, *8*, 3674.

Dr. Martin Hoffmann

(1981)

F-I2 Soft Matter and Functional Materials
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin

Tel: 030 8062 - 43143 (-42308, Fax)
Email: martin.hoffmann@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise:	Polymer- and Colloid Chemistry
Field of Interest:	Anisotropic colloids, polyelectrolytes, microgels, heterogeneous catalysis, depolarized dynamic light scattering

Scientific Development

10/2002 – 08/2007	Study of Polymer- and Colloid Chemistry at the University of Bayreuth
10/2007 – 08/2010	PhD at the University of Bayreuth (Prof. Dr. M. Ballauff): "Synthesis and Characterization of Anisotropic Colloidal Particles"
07/2010 –	Postdoc at the Soft Matter and Functional Materials Institute: Work package leader of FP 7 EU-Project POLYCAT
05/2011 –	Contact person for "Industry Relations" at the Soft Matter and Functional Materials Institute, Helmholtz-Zentrum Berlin, Germany, Prof. Dr. M. Ballauff

Scholarships

10/2002 – 08/2007	Hochbegabtenstipendium BayBFG
05/2008 – 04/2010	Graduiertenstipendium BayEFG

Selected Publications

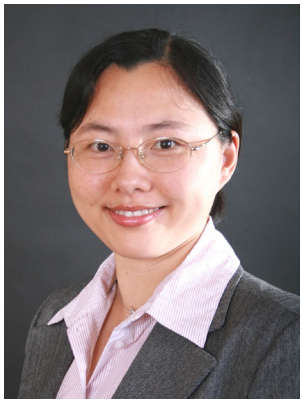
- Schürer, B.; **Hoffmann, M**.; Wunderlich, S.; Harnau, L; Peschel, U.; Ballauff, M. and Peukert, W. Second Harmonic Light Scattering from Spherical Polyelectrolyte Brushes. *J. Phys. Chem. C*. **2011**, *115*, 18302.
- Jiménez, M. L.; Delgado, A. V.; Ahualli, S.; **Hoffmann, M**.; Wittemann, A.; Ballauff, M.: Giant permittivity and dynamic mobility observed for spherical polyelectrolyte brushes. *Soft Matter* **2011**, *7*, 3758.
- **Hoffmann, M.**; Siebenbürger, M.; Harnau, L.; Hund, M.; Hanske, C.; Lu, Y.; Wagner, S.; Drechsler, M.; Ballauff, M.: Thermoresponsive Colloidal Molecules. *Soft Matter* **2010**, *6*, 1125.
- **Hoffmann, M.**; Wagner, C. S.; Harnau, L.; Wittemann, A.: 3D Brownian Diffusion of Submicron-Sized Particle Clusters. *ACS NANO* **2009**, *3*, 3326.

Dr. Yan Lu

(1976)

Nationality: P. R. China

Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und Energie GmbH,
Hahn-Meitner-Platz 1, 14109 Berlin, Germany
Tel.: +49 30 8062 43191; Fax: +49 30 8062 42308
E-Mail: yan.lu@helmholtz-berlin.de



Research interest

- Polymer colloids, including polyelectrolyte brushes and microgels
- Organic/inorganic hybrid colloid particles and their application as catalyst, sensor and solar cells

University Education

09.1994 – 07.1998 Bachelor in Polymer Science and Engineering in College of Material Science and Engineering, Donghua University, Shanghai, P. R. China

09.1998 – 04.2001 M.S. in Material Science in College of Material Science and Engineering, Donghua University, Shanghai, P. R. China

Scientific Degrees

Dr. rer. nat.: (02. 2005) Chemistry, in Institute for Macromolecular Chemistry and Textile Chemistry, Dresden University of Technology, Germany
Supervisor: Prof. Dr. H. J. P. Adler

Scientific Development

04.2005 – 08.2006 Postdoc in the group of Prof. Dr. M. Ballauff, Physical Chemistry I, University of Bayreuth, Germany

09.2006 - 08.2009 Akademische Rätin, Physical Chemistry I, University of Bayreuth, Germany

Since 09.2009 Group leader of Colloid Chemistry, Institute of Soft Matter and Functional Materials, Helmholtz-Zentrum Berlin für Materialien und Energie, Germany

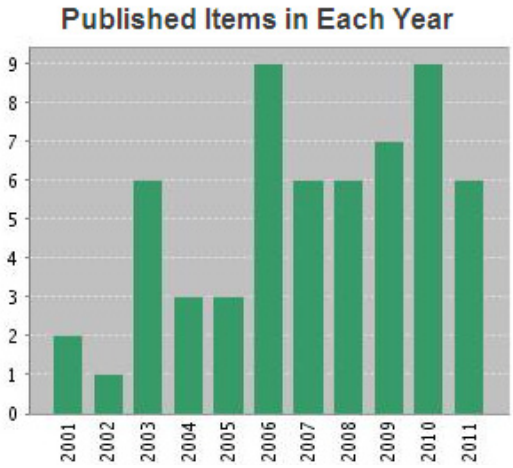
Selected Publications

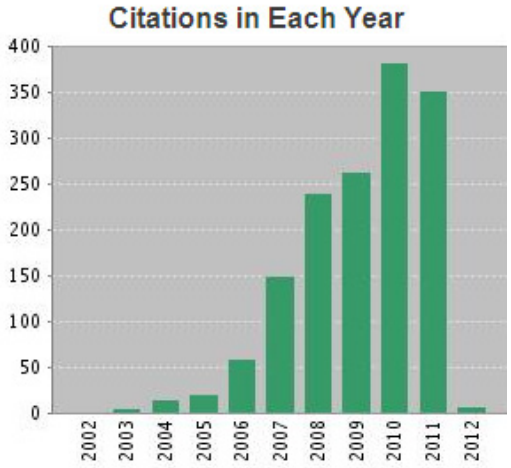
1. Y. Lu, Y. Mei, M. Ballauff*, M. Drechsler, Thermosensitive Core-Shell Particles as Carriers for Ag Nanoparticles: Modulating the Catalytic Activity by a Phase Transition in Networks, *Angew. Chem. Int. Ed.* **45**, 813 (2006).
2. R. Sai Yelamanchili, Y. Lu*, T. Lunkenbein, N. Miyajima, L. Yan, M. Ballauff, J. Breu*, Shaping colloidal rutile into thermally stable and porous mesoscopic titania-balls, *Small* **5**, 1326 (2009).
3. Y. Lu*, J. Yuan, F. Polzer, M. Drechsler, J. Preussner, In-situ Growth of Catalytic Active Au-Pt Bimetallic Nanorods in Thermo-Responsive Core-Shell Microgels, *ACS Nano* **4**, 7078 (2010).
4. Y. Lu, M. Ballauff, Thermosensitive Core-Shell Microgels: From Colloidal Model Systems to Nanoreactors, *Prog. Polym. Sci.* **36**, 767 (2011).
5. S. Wu, J. Dzubiella, J. Kaiser, M. Drechsler, X. Guo, M. Ballauff, Y. Lu*, "Thermosensitive Au-PNIPA Yolk-Shell Nanoparticles with Tunable Selectivity for Catalysis", *Angew. Chem. Int. Ed. DOI: 10.1002/anie.201106515*. (2012)

Award

2005: "APi-Prize" as the best dissertation in 2005 by the German Chemical Society (GDCh) Division of Coatings and Pigments.

2011: "Dr. Hermann-Schnell-Stipendium" by the German Chemical Society (GDCh).





Results found:	58
Sum of the Times Cited [?] :	1493
Sum of Times Cited without self-citations [?] :	1307
Citing Articles [?] :	890
Citing Articles without self-citations [?] :	847
Average Citations per Item [?] :	25.74
h-index [?] :	21

Dr. Uwe Müller

(1967)

Macromolecular crystallography group at BESSY-II
F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Albert-Einstein-Str. 15, 12489 Berlin

Tel: 030 8062-14974 (-14975, Fax)
Email: uwe.mueller@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise:	Macromolecular crystallography using synchrotron radiation
Field of Interest:	Protein structure and functions, Instrumentation of X-ray diffraction beamlines

Scientific Development

Study	1989-1994 Study of Chemistry at the Humboldt University in Berlin
PhD	1999 at Free University Berlin (Prof. Dr. U. Heinemann)
Post-doc	1999-2003 Institut für Kristallographie, Free University Berlin, Prof. Dr. W. Saenger
	2004- 2008 Staff scientist and group leader, BESSY-GmbH, Berlin
	Since 2009 Staff scientist and group leader, Helmholtz Zentrum Berlin

Invited Talks (selection)

"3th Winter school for soft X-ray in macromolecular crystallography", Berlin 2009
"Synchrotron radiation instrumentation 2009", Melbourne 2009

Selected publications

[1] Perl, D., U. Mueller, et al. (2000). *"Two exposed amino acid residues confer thermostability on a cold shock protein."* Nat Struct Biol 7(5): 380-3.

[2] Mueller, U., L. Nyarsik, et al. (2001). *"Development of a technology for automation and miniaturization of protein crystallization."* J Biotechnol 85(1): 7-14.

[3] Heinemann, U., K. Bussow, Mueller U., et al. (2003). *"Facilities and methods for the high-throughput crystal structural analysis of human proteins."* Acc Chem Res 36(3): 157-63.

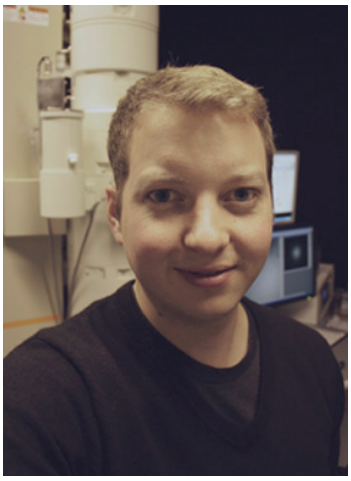
[4] Schonfeld, D. L., R. B. Ravelli, Mueller U., et al. (2008). *"The 1.8-A crystal structure of alpha1-acid glycoprotein (Orosomucoid) solved by UV RIP reveals the broad drug-binding activity of this human plasma lipocalin."* J Mol Biol 384(2): 393-405.

[5] Klein, N., I. Senkovska, Mueller U., et al. (2009). *"A mesoporous metal-organic framework."* Angew Chem Int Ed Engl 48(52): 9954-7.

Dr. Frank Polzer
(1982)

AG TEM, Institute of Physics
Humboldt Universität zu Berlin, Newtonstr.15,
12489 Berlin

Tel: 030 2095-7905 (-7760, Fax)
Email: frank.polzer@physik.hu-berlin.de



Area of Expertise

transmission electron microscopy (TEM) and cryogenic TEM,
reaction kinetics, polymer chemistry, colloidal chemistry and physics, nanoparticle synthesis, hybrid materials

Scientific Development

09/2002 – 06/2007	Study of Polymer- and Colloid Chemistry at the University of Bayreuth
08/2007 – 06/2011	PhD at the University of Bayreuth and the Helmholtz Centre Berlin (Prof. Dr. M. Ballauff): "Composites of Spherical Polyelectrolyte Brushes and Nanoparticles – Synthesis, Characterization and Their Use in Catalysis"
11/2010 – 09/2011	Planning and development of the laboratory for cryogenic transmission electron microscopy of the Joint Laboratory for Structural Research
Since 09/2011	Postdoc at the Institute of Physics, in the TEM group

Selected Publications

- Mei, Yu; Lu, Yan; **Polzer, Frank**; Ballauff, Matthias; Drechsler, Markus. Catalytic Activity of Palladium Nanoparticles Encapsulated in Spherical Polyelectrolyte Brushes and Core-shell Microgels. *Chemistry of Materials* **2007**, *19*, 1062.

  Wunder, Stefanie; **Polzer, Frank**; Lu, Yan; Mei, Yu; Ballauff, Matthias. Kinetic Analysis of Catalytic Reduction of 4-Nitrophenol by Metallic Nanoparticles Immobilized in Spherical Polyelectrolyte Brushes. *Journal of Physical Chemistry C* **2010**, *42*, 7122.

- **Polzer, Frank**; Kunz, Daniel; Breu, Joseph; Ballauff, Matthias. Formation of Ultrathin Birnessite-type Nanoparticles Immobilized on Spherical Polyelectrolyte Brushes. *Chemistry of Materials* **2010**, *22*, 2916.

- **Polzer, Frank**; Heigl, Johannes; Schneider, Christian; Borisov, Oleg; Ballauff, Matthias. Synthesis and Analysis of Zwitterionic Spherical Polyelectrolyte Brushes in Aqueous Solution. *Macromolecules* **2011**, *44*, 1654.

- **Polzer, Frank**; Wunder, Stefanie; Lu, Yan; Ballauff, Matthias. Oxidation of an Organic Dye Catalyzed by MnO_x Nanoparticles. *Journal of Catalysis* **2012**, accepted.

- **Polzer, Frank**; Holub-Krapp, Elizabetha; Rossner, Hermann; Erko, Oleg; Plamper, Felix; Schmalz, Alexander, Müller, Axel H. E.; Ballauff, Matthias. Structural Analysis of Colloidal MnO_x Composites **2012,** submitted.

Dr. Margarita Russina

(1968)

F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin

Tel: 030 8062-43159 (-42308, Fax)
Email: margarita.russina@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise:	Neutron scattering instrumentation, multiplexing methods for pulsed neutron sources, physics of disordered materials
Field of Interest:	Neutron scattering instrumentation, physics of nanostructured energy related materials, physics of disordered matter

Scientific Development

Study	1985- 1992 Study of physics, Moscow Lomonosov University (USSR)
PhD	1994-1998, Technical University Berlin (Prof. Dr. Ferenc .Mezei)
Post-doc	1998-2000 Los Alamos National Laboratory (USA), development of novel neutron scattering methods for instrumentation at pulsed sources
Senior Scientist	2000-2003 Los Alamos National Laboratory (USA), technical leader of Project "Development of High Performance Cold Neutron Spectroscopy at LANSCE" (IN500 Project) Since 2004 Helmholtz Zentrum Berlin, responsible for TOF spectrometer NEAT, scientific leader of NEAT-Upgrade Project

Habilitation at Potsdam University in progress

Selected Scientific Expert, Review or Council Activities, Awards

Member of Scientific Panel, Review activities: Scientific Advisory Committee of European Spallation Neutron Source (ESS) (since 2010); Scientific Advisory Committee of International Collaboration on Advanced Sources (ICANS) (since 2009); On-site panel of Neutron Scattering Science Review Committee of ORNL's High Flux Isotope Reactor (HFIR) and Spallation Neutron Source (SNS) (since 2010); International Advisory Committee of 4th Workshop on Inelastic Neutron spectrometers (WINS), Oak Ridge (USA), 2009; Reviewer of the proposals for neutron beam time at NIST (2003); Reviewer of Phys. Rev. B, Solid State Ionics, Physica B

Awards: Acceptance in Helmholtz Management Academy, Malik Management Center St. Gallen (first year selection), 2008; Los Alamos National Laboratory Achievement Award, September 2001; Young Scientists Award at European Neutron Scattering Conference, Budapest/Hungary, September 1999; Promotionspreis (PHD-Thesis-Award) of Hahn-Meitner-Institute, Berlin/Germany, December 1999

Invited Talks (selection)

"Multiplexing techniques for instruments at pulsed sources", Neutron Instrument Design School, ESS, Lilla Vik, Sweden June 7-17, 2011; "Past, present and future of TOF spectrometer NEAT", Workshop on Trends in Cold Neutron Time-of-Flight Spectroscopy, ILL, Grenoble, France, November 26-28, 2009; "Principle and Implementation of Repetition Rate Multiplication in Neutron Spectroscopy", Workshop on Possible Scientific View from New Neutron Spectroscopy Opportunities in J-PARC, Tokai, Japan, 8-9, July 2009; "Rate Multiplying and pulse shaping chopper systems and the long pulse test at Los Alamos", ESS-Scandinavia Workshop on Long Pulse Spallation Source Instrumentation, Lund (invitations only), April 2006; "Quantum confinement effects for molecular hydrogen in nano-porous materials" , Quasielastic neutron scattering conference, PSI, Villingen, February 8-13, 2009;

Patents:

„Neutronenoptische Bauelementenanordnung zur gezielten spektralen Gestaltung von Neutronenstrahlen oder –pulsen" (Amtl . Aktz. 102 03 591.1 (Germany), date: 23.01.2002; "Neutron-optical components array for the specific spectral shaping of neutron beams or pulses" Patent № US 7030397 B2, April 18, 2006 (USA)

Selected publications

1. M. Russina, F.Mezei "First Implementation of Repetition Rate Multiplication in neutron spectroscopy", J. Nuclear Instrument and Methods, Nuclear Inst. and Methods in Physics Research, A604 (2009) 624
2. M. Russina, Gy. Káli, Zs. Szanto, F. Mezei, "First experimental implementation of pulse shaping for neutron diffraction on pulsed sources". Nuclear Inst. and Methods in Physics Research, 2011, doi:10.1016/j.nima.2011.05.077
3. F.Mezei and M.Russina "Neutron Beam Extraction and Delivery at Spallation Neutron Sources", Physica B, 283 (2000) 318.
4. "Proc. of 3rd Workshop on inelastic neutron spectrometers", Special issue of Journal of Neutron Research, volume 6, 2008, edit. by M. Russina, M. Arai, F.Mezei, H. Muttka, ISSN 1023-8166
5. B. Aoun, M. Gonzalez, J. Ollivier, M. Russina, Z. Izaola, D. Price and M.-L. Saboungi, "Translational and Reorientational Dynamics in Imidazolium-based Liquids," J. Phys. Chem. Lett. 1 (2010) 2503
6. P. Martelli, A.Remhof, A. Borgschulte, P.Mauron, D.Wallacher, E. Kemner, M. Russina, F. Pendolino, and A. Zuettel, "BH_4- Self-Diffusion in Liquid $LiBH_4$", J. Phys. Chem. A, 2010, 114 (37), pp 10117-10121
7. H. A. Hanson, X.Wang, B.B. Maranville, I.K. Dimitrov, C.F. Majkrzak, M.Russina, J. Shi, M. Laver, and X.S. Ling "Growth of Bragg Glass in Edge-Contaminated Vortex Matter", Phys. Rev. B, vol.84 (2011) 014506
8. A. Podlesnyk, M. Russina, A. Furrer, A. Alfonsov, E. Vavilova, V. Kataev, B. Büchner, Th, Strässle, E. Pomjakushina, K.Gonder, D.I. Khomski "Spin-State polaron in lightly hole-doped $LaCoO_3$", Phys.Rev.Lett. 101 (2008) 247603
9. A. Wiedenmann, U. Keiderling, K. Habicht, M. Russina, and R. Gähler ¨Dynamics of field-induced ordering in magnetic colloids studied by new time-resolved Small Angle Neutron Scattering techniques", Phys. Rev. Lett. 97 (2006) 5720
10. M.Russina, F.Mezei and R.Lechner, S.Longeville, B.Urban "Experimental evidence for fast heterogeneous, collective structural relaxation in a supercooled liquid near the glass transition", Phys. Rev.Lett. 84 (2000) pp. 3630-3633

Priv. Doz. Dr. Gerd Schneider

(1963)

F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Albert-Einstein-Str. 15, 12489 Berlin

Tel: 030 8062-13131 (-12114, Fax)
Email: gerd.schneider@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise:	X-ray physics, X-ray imaging, Fourier and X-ray optics, nanotechnology, synchrotron radiation and biophysics
Field of Interest:	X-ray imaging, nano-tomography and X-ray spectromicroscopy applied to biological cells and IT-devices

Scientific Development

Study	1983 - 1988 Study of chemistry at the University of Göttingen
PhD	1992 University of Göttingen (Prof. Dr. G. Schmahl)
	1992 Ernst-Eckhard-Koch award
Post-doc	1992 - 1995 Institute for X-ray Physics, University of Göttingen,
	1996 - 1999 Research Assistant (C1), Institute for X-ray Physics, University of Göttingen
	1999 Habilitation in Physics, University of Göttingen
	2000 Heisenberg Fellowship of the DFG
Senior Scientist	2000 - 2002 Lawrence Berkeley National Laboratory
	2002 2nd position on list C4-professorship University of Göttingen
	2003 - 2008 Group leader, BESSY m.b.H., Berlin
	2005 Priv.Doz. at Humboldt Universität zu Berlin
	2009 Head of Department for Microscopy, Helmholtz Zentrum Berlin
	2010 Group leader Microscopy Helmholtz Zentrum Berlin

Selected Scientific Expert, Review or Council Activities, Awards

Ernst-Eckhard-Koch award 1992, Heisenberg fellowship 2000, Editorial Advisory Board of Current Nanoscience (since 2005), Member of the International Program Committee of the International Conference on X-ray Microscopy (since 2005), Reviewer for Nature, Applied Physics Letters, Ultramicroscopy, Optics Express, referee for the Körber-Stiftung

Invited Talks (selection)

2009 NSLS/CFN Joint Users' Meeting, Workshop 2: The Cold, Soft Truth: Cryo Systems for Studying Soft Materials, Brookhaven National Laboratory, May 18, 2009

SCANDEM 2009-Annual Meeting of the Nordic Microscopy Society University of Iceland, Reykjavík, Iceland, 2009

First International Symposium on Structural Systems Biology, Hamburg, September 24- 25, 2009

Frontiers in Optics, San Jose, USA 2009

Seminar at SOLEIL Synchrotron, November 3, 2009, France

Workshop on Correlative Microscopy, Oxford 2010; England

Microscience 2010, London 2010; England

Swedish-German Workshop on In Situ X-ray Techniques, 6-7 October 2011, Rosersberg Castle, Sweden

Workshop on Stress Management for 3D IC's using Through Silicon Vias, 12. Oct. 2011, Dresden

8th Münster Conference on Single Cell and Molecular Analysis, 16-17. Nov. 2011, Münster

Selected publications

[1] G. Schneider, S. Rehbein, S. Werner, *Volume Effects in Zone Plates* in: Modern Developments in X-Ray and Neutron Optics Springer Series in Optical Sciences, Springer Berlin/Heidelberg **137** (2008), 137-171

[2] S. Rehbein, S. Heim, P. Guttmann, S. Werner, G. Schneider, *Ultrahigh-resolution soft-x-ray microscopy with zone plates in high orders of diffraction*, Phys. Rev. Lett. **103**, (2009) 110801

[3] G. Schneider, P. Guttmann, S. Heim, S. Rehbein, F. Mueller, K. Nagashima, J.B. Heymann, W.G. Müller, J.G. McNally, *Three-dimensional cellular ultrastructure resolved by X-ray microscopy,* Nature Methods **7** (2010), 985-987

[4] P. Guttmann, C. Bittencourt, S. Rehbein, P. Umek, X. Ke, G. Van Tendeloo, C. P. Ewels and G. Schneider, *Nanoscale spectroscopy with polarized X-rays by NEXAFS-TXM*, Nature Photonics 6 (2012), 25-29

[5] G. Schneider, P. Guttmann, S. Rehbein, S. Werner, R. Follath, *Cryo X-ray microscope with flat sample geometry for correlative fluorescence and nanoscale tomographic imaging*, J. Struct. Biol. 177 (2012), 212-223

Dr. Sebastian Seiffert (Dipl.-Chem.)

(1979)

F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin

Tel.: +49 30 8062 42294
Email: sebastian.seiffert@helmholtz-berlin.de
seiffert@chemie.fu-berlin.de

Area of Expertise

Polymer Chemistry, Polymer Physics, Physical Chemistry, Chemical Engineering

Scientific Career

Since 01/2011	Junior research group leader at Helmholtz-Zentrum Berlin and FU Berlin, Germany • Liebig Fellow of the Fund of the German Chemical Industry • Research on *Supramolecular and Stimuli-Sensitiveve Polymer Gels* • Lecturer on *Polymer Physics*
01/2009–12/2010	Postdoctoral fellow with Prof. D. A. Weitz, Dpt. of Physics and SEAS, Harvard University, Cambridge, Massachusetts, U.S.A. • Research Fellowship by the German Acad. of Sciences Leopoldina • Subject of Research: *Functional Polymer Microgels*
01/2008–12/2008	Postdoctoral fellow with Prof. W. Oppermann, Institute of Physical Chemistry, Clausthal University of Technology, Germany • Subject of Research: *Structure and Dynamics in Polymer Gels* • Lecturer on *Structure and Dynamics in Polymer Systems*
12/2007	PhD degree, Clausthal University of Technology, Germany • Predicate: summa cum laude • Thesis "*Structure and Tracer Dynamics in Polyacrylamide Gels*"
08/2004–12/2007	PhD student with Prof. W. Oppermann, Institute of Physical Chemistry, Clausthal University of Technology, Germany • Subject of Research: *Structure and Dynamics in Polymer Gels*
07/2004	Diploma in Chemistry, Clausthal University of Technology • Degree: Dipl.-Chem; passed with distinction (mit Auszeichnung) • Diploma thesis "*Diffusion of Linear Polyacrylamide Chains in Semidilute Systems*"
10/1999–08/2004	Study of Chemistry at Clausthal University of Technology

Awards and Grants

Since 01/2011	Liebig Fellowship by the Fund of the German Chemical Industry for the establishment of a junior research group at Helmholtz Zentrum Berlin and FU Berlin
01/2009–12/2010	Research fellowship by the German National Academy of Sciences Leopoldina for a biennial postdoctoral stay at Harvard University, Cambridge, Massachusetts, U.S.A.
10/2009	Dissertation Award by the Society of Friends of Clausthal University of Technology
06/2008	Offer of a Postdoc Scholarship by the German Research Foundation (declined in favor of scholarship by the German Academy of Sciences Leopoldina)
02/2002	Nominee for scholarship by the German National Academic Foundation
12/2001	Book award by the German Chemical Society (GDCh) for outstanding performances as an undergraduate

Selected Publications

S. Seiffert and J. Sprakel, “Physical chemistry of supramolecular polymer networks.” *Chem. Soc. Rev.* **2012**, *41*, 309–330.

S. Seiffert and D. A. Weitz, “Controlled Fabrication of Polymer Microgels by Polymer-Analogous Gelation in Droplet Microfluidics.” *Soft Matter* **2010**, *6*, 3184–3190.

S. Seiffert, J. Thiele, A. R. Abate and D. A. Weitz, “Smart Microgel Capsules from Macromolecular Precursors.” *J. Am. Chem. Soc.* **2010**, *132*, 6606–6609.

S. Seiffert and W. Oppermann, “Diffusion of Linear Macromolecules and Spherical Particles in Semidilute Polymer Solutions and Polymer Networks.” *Polymer* **2008**, *49*, 4115–4126.

S. Seiffert and W. Oppermann, “Systematic Evaluation of FRAP Experiments Performed in a Confocal Laser Scanning Microscope.” *J. Microsc.* **2005**, *220*, 20–30.

Dr. Roland Steitz

(1960)

F-I2 Soft Matter and Functional Materials,
Helmholtz-Zentrum Berlin für Materialien und
Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin

Tel: 030 8062-42149 (-42308, Fax)
Email: steitz@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise:	Physics of soft matter interfaces
Field of Interest:	Chemistry and physics of amphiphiles, scattering methods

Scientific Development

Study	1978- 1988 Study of chemistry at the University of Mainz
PhD	1993 at the University of Mainz (Prof. Dr. H. Möhwald)
Post-doc	1994-1995 Dept. Chemistry, University of Queensland, Brisbane, Australia, Dr. G. Barnes
	1995- 2000 Junior Scientist, Technische Universität Berlin
	2000- 2004 Junior Scientist, Max-Planck Institut für Kolloid und Grenzflächenforschung, Golm
Senior Scientist	Since 2004, Institute for Soft Matter and Functional Materials, HZB, Berlin

Habilitation at TU Berlin in progress

Selected Scientific Expert, Review or Council Activities, Awards

Member of Scientific Panel: Sub-Committee 9, Institut Laue-Langevin, Grenoble, France (2003-2006), Member of BENSC Scientific Panel, Hahn-Meitner-Institut, Berlin (2003), Member of German Committee for Research with Neutrons, KFN (2005-2011)

Invited Talks (selection)

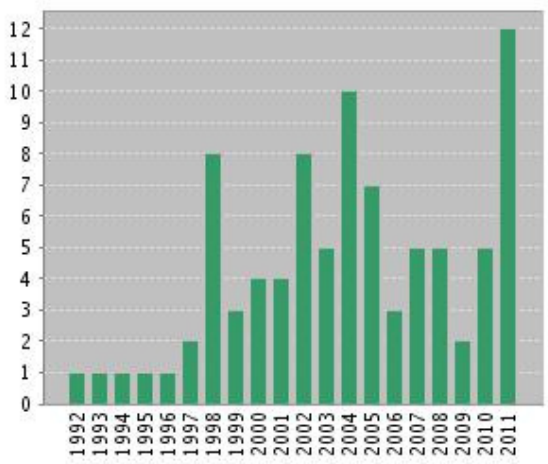
"Physikalisches Kolloquium", Universität des Saarlandes, Saarbrücken 2005; "52nd AVS International Symposium", Boston, 2005; "ADAM Workshop" Ruhr-Universität, Bochum 2006; "2nd BENSC Adsorption Workshop HMI, Berlin 2007; „Workshop zum Thema Streumethoden, SPP 1273 Kolloidverfahrenstechnik", Bayreuth 2008; "GISAS 2009", Satellite Conference of SAS 2009, Hamburg 2009; Department of Physics and Astronomy, Uppsala University, Sweden 2010; "DPG-Frühjahrstagung", TU-Dresden, 2011; "Workshop on off-specular neutron scattering" Universite Libre de Bruxelles, Brussels 2012

Selected publications

1. M. Kreuzer, T. Kaltofen, R. Steitz, B. H. Zehnder, R. Dahint, *Pressure cell for investigations of solid-liquid interfaces by neutron reflectivity,* Review of Scientific Instruments 2011, **82** (2), 023902-7
2. F. Evers, R. Steitz, M. Tolan, C. Czeslik, *Analysis of Hofmeister effects on the density profile of protein adsorbates - a neutron reflectivity study,* Journal of Physical Chemistry B 2009, **113**, 8462-8465
3. M. Wolff, R. Steitz, P. Gutfreund, N. Voss, S. Gerth, M. Walz, A. Magerl, H. Zabel, *Shear Induced Relaxation of Polymer Micelles at the Solid−Liquid Interface*, Langmuir 2008, **24**, 11331
4. V. Papaefthimiou, R. Steitz, G. H. Findenegg, *Schaltbare Oberfläche – Responsive Polymerschichten* Chemie in unserer Zeit 2008**, 42**, 102-115
5. C. Czeslik, G. Jackler, R. Steitz and H.-H. von Grünberg, *Protein Binding to Like-Charged Polyelectrolyte Brushes by Counterion Evaporation*, J. Phys. Chem. B 2004, **108**, 13395.
6. R. Steitz, T. Gutberlet, T. Hauß, B. Klösgen, R. Krastev, S. Schemmel, A. C. Simonsen and G. H. Findenegg, *Nanobubbles and Their Precursor Layer at the Interface of Water Against a Hydrophobic Substrate*, Langmuir 2003, **19**, 2409
7. R. Steitz, W. Jaeger, R. v. Klitzing, *Influence of charge density and ionic strength on the multilayer formation of strong polyelectrolytes*, Langmuir 2001, **17**, 4471
8. R. Steitz, V. Leiner, R. Siebrecht and R. v. Klitzing, *Influence of the ionic strength on the structure of polyelectrolyte films at the solid/liquid interface*, Colloids and Surfaces A 2000, **163**, 63
9. M. Tarabia, H. Hong, D. Davidov, S. Kirstein, R. Steitz, R. Neumann, Y. Avny, *Neutron and X-ray reflectivity studies of self-assembled heterostructures based on conjugated polymers*, J. Appl. Phys. 1998, **83**, 725-732

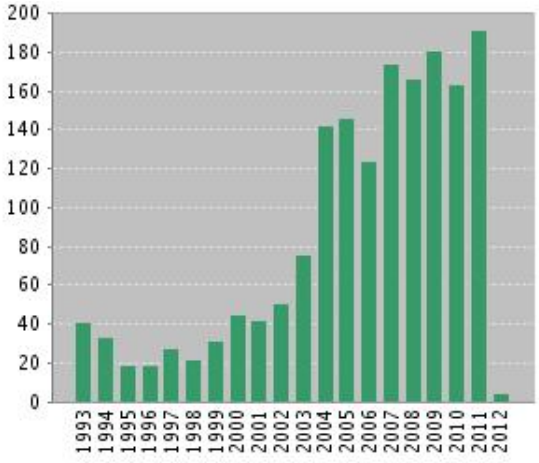
Citation report Web of Science[SM] 13.02.2012

Published Items in Each Year



Years

Citations in Each Year



Years

Results found:	96
Sum of the Times Cited:	1734
Average Citations per Item:	18.06
h-index:	23

Dr. Manfred S. Weiss (1963)

Macromolecular crystallography group at BESSY-II
F-I2 Soft Matter and Functional Materials
Helmholtz-Zentrum Berlin für Materialien und Energie GmbH
Albert-Einstein-Str. 15
D-12489 Berlin

Tel: 030 8062-13149 (-14975, Fax)
Email: manfred.weiss@helmholtz-berlin.de

Area of Expertise and Field of Interest

Area of Expertise: Macromolecular crystallography using synchrotron radiation

Field(s) of Interest: Protein structure and function, macromolecular crystallization, instrumentation of X-ray diffraction beamlines

Scientific Development

Study 1982-1989: Study of Chemistry at the Albert-Ludwigs-Universtät Freiburg i. Br. and the University of Massachussetts at Amherst, USA.

Ph.D. 1989-1992: at Albert-Ludwigs-Universtät Freiburg (Prof. Dr. G. E. Schulz)

Post-doc 1992-1996 Molecular Biology Institute, University of California at Los Angeles, USA (Prof. Dr. D. Eisenberg)

1996-2001 Senior Research Assistant at the Institute for Molecular Biotechnology, Jena (Prof. Dr. R. Hilgenfeld)

2001-2009 Team Leader at the EMBL Hamburg Outstation

2009-present Staff scientist, Helmholtz Zentrum Berlin

Other Activities

- Member of the National Committee of the DGK (since 04/06).
- Co-editor of Acta Crystallographica Section D (since 08/02).
- Co-editor of Acta Crystallographica Section F (since 04/07).
- Section Editor of Acta Crystallographica Section F (since 08/08).
- Member of the IUCr Commission on Crystallographic Teaching (since 08/11).

Invited Talks (selection)

Conference: European Crystallographic Meeting ECM-26, 31.08.-02.09.2010, Darmstadt, Germany

Conference: American Crystallographic Association (ACA) Meeting 2010, 24.-30.07.2010, Chicago, USA

Course: *Training Methods for Macromolecular Crystallography. From Measurement to Model - 2009*, M2M-9, 21.-28.10.2009, EMBL Hamburg Outstation, Hamburg, Germany.

EMBO-Course: *Exploiting Anomalous Scattering in Macromolecular Structure Determination*, 15.-22.06.2009, ESRF Grenoble, France.

3rd Winter School on *Soft X-rays in Macromolecular Crystallography*, 18.-20.02.2009, Berlin, Germany.

Conference: International Union of Crystallography (IUCr) Conference 2008, 23.-30.08.2008, Osaka, Japan.

Workshop: ISRTMSF2008, 07.-11.01.2008, University of Madras, Guindy Campus, Chennai, India.

Selected publications

T. Werther, A. Zimmer, G. Wille, R. Golbik, **M. S. Weiss** & S. König (2010). New Insights into Structure-Function Relationships of Oxalyl-CoA Decarboxylase from *Escherichia coli*. FEBS J. 277, 2628-2640.

M. J. Belousoff, C. Davidovich, E. Zimmerman, Y. Caspi, I. Wekselman, L. Rozenszajn, T. Shapira, O. Sade-Falk, L. Taha, A. Bashan, **M. S. Weiss** & A. Yonath (2010). Ancient Machinery Embedded in the Contemporary Ribosome. Biochem Soc Trans. 38, 422-427.

R. Janowski, G. Kefala & **M. S. Weiss** (2010). The structure of Dihydrodipicolinate Reductase (DapB) from *Mycobacterium tuberculosis* in Three Crystal Forms. Acta Cryst. D66, 61-72.

L. Schuldt, S. Weyand, G. Kefala & **M. S. Weiss** (2009). The Three-dimensional Structure of a Mycobacterial DapD Provides Insights into DapD Diversity and Reveals Unexpected Particulars About the Enzymatic Mechanism. J. Mol. Biol. 389, 863-879.

R. Janowski, S. Panjikar, A. Nasser Eddine, S. H. E. Kaufmann & **M. S. Weiss** (2009). Structural Analysis Reveals DNA binding Properties of Rv2827c, a Hypothetical Protein from *Mycobacterium tuberculosis*. J. Struct. Funct. Genom. 10, 137-150.

C. Mueller-Dieckmann, S. Panjikar, A. Schmidt, S. Mueller, J. Kuper, A. Geerlof, M. Wilmanns, R. K. Singh, P. A. Tucker & **M. S. Weiss** (2007). On the Routine Use of Soft X-Rays in Macromolecular Crystallography, Part IV - Efficient Determination of Anomalous Substructures in Bio-Macromolecules Using Longer X-ray Wavelengths. Acta Cryst. D63, 366-380.

C. Mueller-Dieckmann, S. Kernstock, M. Lisurek, J. P. von Kries, F. Haag, **M. S. Weiss** & F. Koch-Nolte (2006). The Structure of Human ADP-Ribosylhydrolase 3 (ARH3) Provides Insights into the Reversibility of Protein ADP-Ribosylation. Proc. Natl. Acad. Sci. USA 103, 15026-15031.

Soft Matter and Functional Materials

Table of content

4. Research

Macromolecular Crystallography (MX)

Phase determination using the anomalous signal from sulphur atoms

Phase determination using the UV-light induced radiation damage

Diffraction-based screening to rapidly characterize biological crystals in their native environment

Controlled crystal dehydration using the HC1c device installed at BL14.3

Structure and function of protein complexes from the spliceosome

Xenobiotic reductase A of Pseudomonas putida

Biological degradation of halogenated organic pollutant: Enzymes, Structures & Mechanisms

Biophysics

Time-resolved protein dynamics

Interactions between Proteins and Colloidal Particles

Dynamics of phospholipid membranes influenced by the Alzheimer's disease amyloid-β peptide analogue

Interaction of a γ-secretase modulator with model lipid membranes

Interaction strength between proteins and polyelectrolyte brushes: A small-angle X-ray scattering study

Intracellular localization of docosahexaenoic acid (DHA) in human B cells

Colloid Physics

Anomalous small-angle X-ray scattering – a precise experimental technique for the quantitative analysis of nano-scaled phases in chemistry and physics

Analysis of the critical Casimir effect in binary liquid mixtures by V-SANS

Correlation of nanostructure and photoconductivity in amorphous Silicon-Germanium alloys – solar- cell materials analyzed by anomalous small-angle X-ray scattering

Analysis of interaction of thermo-responsive colloidal dumbbells by V-SANS

The colloidal stability of spherical polyelectrolyte brushes

Yielding of a concentrated suspension observed by FT-Rheology: Comparison with the Mode Coupling Theory

V-16 – A new SANS instrument for soft-matter research

Influence of charge density on bilayer bending rigidity in lipid vesicles: a combined dynamic light scattering and neutron spin-echo study

Structural investigations aiming core-multishell nanoparticles

Polyethylene nanocrystals – small-angle X-ray studies

Small Angel Scattering on Protein and Lipid Systems

Interfaces

The swelling/stability effect of hyaluron on a lipid multilayer system

Lubrication in natural joints – a sheer dependence study

Functional Interfaces – brushes and pressure

Effect of uniaxial Strain of Polyelectrolyte Multilayers

Anisotropic Fluids at Solid Interfaces: Ionic Liquids

Influence of Trehalose on the Nano-Structure of Lipid Membranes

V18 Bioref – a versatile tool for surface/interface characterizations

Fourier transform infrared spectroscopy (FTIR) at solid-liquid interfaces

Pressure cell for investigations of solid-liquid interfaces by neutron reflectivity

Colloid Chemistry

Spherical Polyelectrolyte brushes as “Nanoreactors” for Metal Nanoparticles or Nanoalloys

Spherical polyelectrolyte brushes as “nanoreactor” for well-defined Crystalline TiO_2 Nanoparticles

Formation of Ultrathin Birnessite-Type Nanoparticles Immobilized on cationic Spherical Polyelectrolyte Brushes

Kinetic Studies of Reduction of 4-Nithrophenol using Metal Nanoparticles immobilised in Spherical Polyelectrolyte Brushes as Catalyst

Thermosensitive Core-Shell Microgels as active “Nanoreactors” for Metal nanoparticles

Dynamics of Colloidal Suspensions of Anisometric Particles

Plasmonic Au-based nanocomposite particles: synthesis, characterization and application

In-situ Growth of Catalytic Active Au-Pt Bimetallic Nanorods in Thermo-Responsive Core-Shell Microgels

Thermosensitive Au-PNIPA Yolk-Shell Nanoparticles with Tunable Selectivity for Catalysis

X-Ray Microscopy

Development of Diffractive Optics for High-Resolution X-Ray Imaging

Correlative 3-D Microscopy for Life Sciences

Materials science applications

X-ray optical methods and instrumentation for advanced X-ray microscopy

Nano-Tomography of Cells

NEXAFS/cryo-TXM on colloidal hybrid structures

Soft Matter Theory

SFB-951 project A1: Exploring molecular-structure formation of hybrid inorganic/organic systems for opto-electronics (HIOS) by all-atom molecular dynamics computer simulations

Modelling protein adsorption using atomistic and coarse-grained computer simulations

Structure and Dynamics of Colloidal Dumbbell Suspensions

Salt Induced Hydrophobic Collapse of Biopolymers

Time-of-Flight Spectroscopy

NEAT upgrade project

Multiplexing technique: adjustable short pulses at long-pulse spallation source

Confinement effects on moleacular hydrogen dynamics in ice-based clathrates

Polymer Physics

Industry Relations

POLYCAT: Modern polymer-based catalysts and microflow conditions as key elements of innovations in fine chemical syntheses

Complex characterizations of nanocolloids by hyphenation of various separation techniques with inductively coupled plasma mass spectrometry

5. CVs

Edited by Dr. Nikoline Hansen and Dr. Martin Hoffmann

Helmholtz-Zentrum Berlin für Materialien und Energie
Soft Matter and Functional Materials
Hahn-Meitner-Platz 1, 14109 Berlin

www.helmholtz-berlin.de

+ 49 30 8062 43074

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