CPP 4 New Experimental Techniques

Time: Monday 14:00–17:45 Room: ZEU Lich

Invited Talk

CPP 4.1 Mon 14:00 ZEU Lich

Optical tweezers - novel tools in nanophysics — •Friedrich Kremer, Christof Gutsche, Kati Kegler, Mathias Salomo, Joerg Reinmuth, and Wiktor Skokow — University of Leipzig, Institute of Experimental Physics I

Optical tweezers are microscopic tools with extraordinary resolution.(resolution in space: ± 1 nm, resolution in force: ± 50 fN) In the talk three different experiments will be discussed: 1.) Measurement of the force-extension dependence of single chains of double stranded DNA having a length between 1000 base pairs (bp) to 6000 bp. It is shown that the wormlike chain model fits the data. 2.) Measurement of forces of interaction between single blank and DNA-grafted colloids. The forces are measured in two directions, parallel and perpendicular to the axis between the two colloids under study. Bringing the DNA-grafted colloids in close contact (< 100 nm for 4000 bp DNA) enables one to measure directly the forces necessary to interdigitate the DNA-brushes. Under these conditions frictional stick-slip phenomena take place, when the colloids are moved perpendicularly to its central axis. 3.) Measurement of the flow resistance of single colloids grafted with DNA of lengths between 1000 bp to 6000 bp. The experiments can be described by Stokes law with an effective hydrodynamic radius being dependent on the flow velocity, the length, the grafting-density and the ionic strength of the surrounding medium.

CPP 4.2 Mon 14:30 ZEU Lich

Gold Nanorods as Novel Nonbleaching Plasmon-Based Sensors for Molecular Orientations and Local Refractive Index Changes — •Jan Becker and Carsten Sönnichsen — Nanobiotechnology group, Institute for Physical Chemistry, University of Mainz, Germany (www.nano-bio-tech.de)

Gold nanorods of typical dimensions of 25nm x 60nm can be observed in a darkfield microscope because of their strong light scattering at the plasmon resonance. The scattered light of these gold rods is strongly polarized along their long axis making them an ideal orientation probe. We study the rotations of single gold nanorods weakly bound to a glass surface by observing the intensity fluctuations in polarized light. We observe some fast rotating rods (10ms time scale) and some very slow rotating rods likely trapped in shallow surface potentials.

Furthermore, the wavelength of the plasmon depends on the volume and aspect ratio of the gold nanorod, but also on the refractive index of the local environment. We study this plasmon shift for different particle shapes to find the optimal local refractive index sensor using a novel automated acquisition setup. Nanorods are found to be more sensitive than spherical particles, triangles, cubes and hollow spheres.

CPP 4.3 Mon 14:45 ZEU Lich

FTIR-Goniometry — •Patrick Koelsch, Michael Tammer, and Friedrich Kremer — University of Leipzig, Faculty of Physics and Earth Science, Linnestrasse 5, 04103 Leipzig

Fourier Transform Infrared (FTIR)-Goniometry is a novel spectroscopic tool in which the principle of goniometry (as known from x-ray scattering) is combined with (polarized) IR spectroscopy. In practice a thin (μm thick) sample is intentionally inclined in adjustable spatial directions with respect to the optical axis of the spectrometer. An algorithm will be presented to deduce the mean orientation and the distribution of the different molecular moieties in the sample from the measured transmission. Thereby advantage is taken from the specificity of the IR spectral region as a "fingerprint" regime.

This offers a manifold of novel perspectives for molecular physics and materials science. Examples will be discussed.

CPP 4.4 Mon 15:00 ZEU Lich

Transitions in Nanometer thin films - Investigations by AC-Chip calorimetry — •Heiko Huth, Alexander Minakov, and Christoph Schick — University of Rostock, Institute of Physics, Universitätsplatz 3, 18051 Rostock, Germany

Calorimetry is known as a very powerful tool for the characterization of a wide variety of materials and their transitions. The combination of silicon technology and calorimetry opens up new possibilities in this research area as demonstrated recently (1). Based on a differential AC-calorimeter we show an improved experimental setup combining the advantages of the different methods. The measurements are done at slow scanning or at constant bath temperature. The frequency chosen provides a well defined time scale of the experiment. In several cases, e.g. at glass transition, a direct comparison with results from other dynamic methods like dielectric spectroscopy is possible. Due to the differential setup we achieve a sensitive in the pico Joule per Kelvin range allowing to measure samples below one nanogram and consequently films down to 1 nm thickness (2, 3). Because of the small total heat capacity (addenda + sample) not only a high sensitivity is achieved but AC measurements at relative high frequencies are possible too. The capabilities are demonstrated by different nanometer thin polymeric films.

[1] D. W. Denlinger; E. N. Abarra; K. Allen; P. W. Rooney; M. T. Messer; S. K. Watson; F. Hellman, Rev. Sci. Instrum. 65 (1994) 946
[2] H. Huth, A. Minakov, C. Schick, Netsu Sokutei 32 (2005) 69
[3] Lupascu V., Huth H., Schick C. and W"ubbenhorst M. Thermochim. Acta 432 (2005) 222

CPP 4.5 Mon 15:15 ZEU Lich

Visualizing the metal centers in metallocomplexes by scanning tunneling spectroscopy — •M.S. Alam[1], V. Dremov[1], P. Müller[1], R. Alsfasser[2], U. Kortz[3], M. Ruben[4], L.K. Thompson[5], and J.M. Lehn[6] — [1]Physikalisches Institut III, Universität Erlangen — [2]Institut für anorganische Chemie, Universität Freiburg — [3]School of Engineering and Sciences, International University Bremen — [4]Institut für Nanotechnologie, FZ Karlsruhe — [5]Dept. of Chemistry, Memorial University, St. John*s, NL, Canada — [6]ISIS, Université Louis Pasteur, Strasbourg, France

As the contrast in STM images mixes both topographic and electronic effects in a complicated way, mapping of complex molecules is not straightforward. However, scanning tunneling spectroscopy (STS) measurements reveal direct information about the energy levels close to moleculés Fermi level. Using our home built STM working under ambient conditions, we succeeded to combine high resolution topography mapping with simultaneous current-voltage characteristics measurements on single molecules deposited on HOPG surfaces.We present our recent results on Co [2X2], Mn [3X3] grid-type molecules, Cu20 wheel-shaped polyoxoanions, as well as on Cu coordination polymers. In our STS measurements we found a rather large signal at the expected positions of the metal centers in our molecules, i.e. the location of the individual metal ions in their organic matrix is directly addressable by STS even if these ions are covered by the organic ligands.

— 15 min. break —

CPP 4.6 Mon 15:45 ZEU Lich

Energy Dispersive Small Angle X-Ray Scattering — •Tushar Sant[1], Wolfram Leitenberger[2], Tobias Panzner[1], and Ullrich Pietsch[1] — [1]Institute for Physics, University of Siegen, Walter Flex Strasse 3, D-57068 Siegen, Germany. — [2]Institute for Physics, University of Potsdam, Am Neuen Palais 10, D-14469 Potsdam, Germany.

Small Angle X-ray Scattering is well known technique for characterizing polymers, colloids and biological samples. We performed energy dispersive small angle X-ray scattering (EDSAXS) experiments using white synchrotron radiation at Energy Dispersive Reflectivity (EDR) beamline at BESSY II. We measured EDSAXS spectra for Gold nanoparticles with diameter of about 12 nm and 40 nm solved in water. The extracted colloidal radius taken from EDSAXS spectra agree well with these obtained by monochromatic SAXS measurements at an in-house set-up. The main advantage of EDSAXS technique is the most reduced time consumption which required 3 minutes at EDR but 3 hrs for monochromatic measurement. This is useful for the systematic investigation of large series of samples or study of slow dynamical processes. The specific experimental conditions at BESSY II limit the accessible q range of investigation. This in turn puts the limit for the measurable size of the particles.

CPP 4.7 Mon 16:00 ZEU Lich

High resolution setups at BW4 - from microfocus to USAXS — •Stephan V. Roth, Ralph Döhrmann, Martin Dommach, Christian Schroer, Marion Kuhlmann, and Rainer Gehrke — HASYLAB at DESY, Notkestr. 85, D-22603 Hamburg

The wiggler beamline BW4 at HASYLAB is dedicated to ultra-small-

angle x-ray scattering (USAXS) in material science. After ten years of successful operation major upgrading and refurbishment has been performed, starting in 2003. We describe the recent upgrade of the USAXS-camera, the available SAXS setups and the present status of BW4 on the basis of standard samples and recent experiments. In brief, the key improvements at BW4 are as follows: 1) The maximum USAXS resolution - defined by the size of the beam stop - is $d_{max} > 1\mu$m at a sample-to-detector distance $L_{SD} = 13$m. 2) In grazing incidence USAXS (GIUSAXS) the reolution has been pushed well beyond $d_{max} > 3\mu$m. 3) A new SAXS setup using a newly designed piezo-driven portable slit is available. This allows to optimize intensity and resolution for $L_{SD} \leq 4$m. 4) A new microfoucs option has been established using Beryllium compound refractive lenses. Currently a moderate microfocus of 30x17μm^2 can be achieved. This new option allows for scanning applications like μSAXS and μGISAXS with the sample size reduced by two orders of magnitude.

CPP 4.8 Mon 16:15 ZEU Lich

Periodic NMR excitation in very thin slices: Interplay of diffusion and relaxation — •Achim Gädke and Nikolaus Nestle — TU Darmstadt, Institute of condensed matter physics, Hochschulstr. 6, D-64289 Darmstadt, Germany

NMR experiments involving thin excited slices are of interest both for conventional micro-MRI and for mechanically detected magnetic resonance. Especially in the latter case, the thickness of the excited slices may be well below 1 μm. In samples containing fast-diffusing liquid components such as water, the diffusive exchange of spin magnetization between the excited slice and its surroundings on the time scale of the NMR experiments plays a major role. Some time ago, we have studied the influence of the diffusion balance on the apparent longitudinal relaxation behaviour measured in a saturation-recovery experiment. This study suggests that in a 1 μm slice with a Gaussian profile, the apparent longitudinal relaxation time of water may be below 1 ms. In this experiment, full spin relaxation was possible between two excitation cycles. In micro-MRI or in mechanically detected NMR, by contrast, the excitation is periodic. In this case, the diffusive magnetization balance between the excited slice and its surroundings needs considering excited magnetization from previous excitation cycles, too. This diffusion balance will also depend on the presence of diffusion barriers and relaxation sinks outside the excited slice. Results from both simulation studies and experiments concerning these two effects are presented, and their implications micro-MRI and mechanically detected NMR are discussed.

CPP 4.9 Mon 16:30 ZEU Lich

Visualization of liquid water in PEM Fuel Cells with Neutron Radiography — •Ingo Manke[1,2], Nikolay Kardjilov[2], Christoph Hartnig[3], André Hilger[2,4], Markus Strobl[4], Manfred Grünerbel[3], Wolfgang Treimer[4], Werner Lehnert[3], and John Banhart[1,2] — [1]Technische Universität Berlin, Fakultät 3 — [2]Hahn-Meitner-Institut Berlin, SF 3 — [3]Centre for Solar Energy and Hydrogen Research (ZSW) — [4]University of Applied Science (TFH), FB 2

A key issue in the development of Polymer Electrolyte Membrane Fuel Cells (PEMFC) is the optimization of the mass transport, i.e. the transport of liquid water, in the gas diffusion layers and the gas channels of the flow field. The water is produced by conversion of hydrogen fuel and oxygen into electrical energy. However, the production of too much water can disturb the gas flows and causes a breakdown in the output power. Neutron Radiography (NR) is a unique experimental technique to study the two-phase flow inside operating PEMFCs. Neutrons penetrate quite thick metal components and at the same time they are highly sensitive to hydrogen containing materials. The accumulation of water was investigated under different operating conditions. Beginning from *dry* and going to *wet* conditions, time and location of the water accumulation were recorded, analyzed and quantified by NR. First liquid water droplets develop in the gas diffusion layers. After some time water drops appear at the side of the gas channels in the flow field. The drop sizes are increasing until the whole channel is filled and the gas flow inside the whole channel is filled and the gas flow inside the corresponding channels is blocked.

CPP 4.10 Mon 16:45 ZEU Lich

3-d Cylindrical Pulsed X-Band ESR-Imaging — •Michael Glied[1,2], Malte Drescher[2], and Elmar Dormann[2] — [1]now at 1. Physikalisches Institut, Universität Stuttgart, D-70550 Stuttgart, Germany — [2]Physikalisches Institut, Universität Karlsruhe (TH), D-76128 Karlsruhe, Germany

ESR-Imaging is a technique using similar principles to the established NMR-imaging; however, the fast electron spin relaxation time constants are an experimental challenge. Spin echo backprojection reconstruction imaging in the X-band using a static magnetic field gradient is the favourable application for material science where temperature variation in metal cryostat is necessary. Nevertheless, studying quasi-one dimensional organic conductors only a pulsed phase encoding gradient can be tolerated in the conduction direction in order to avoid imaging artefacts caused by electron spin diffusion.

We present an appropriate three dimensional imaging scheme, combining static gradient backprojection reconstruction and pulsed gradient fourier imaging to scan the reciprocal space in cylindrical coordinates and obtain three dimensional images of various physical parameters like spin density, relaxation time or defect concentration.

Conduction electron density images with a volume element size of $13 \times 13 \times 17\mu m^3$ as well as images of the spin lattice relaxation time, obtained by inversion recovery, with a volume element size of $13\times13\times68\mu m^3$ are presented for fluoranthene radical cation salt single crystals of typical sizes of $0.4 \times 0.4 \times 1mm^3$.

CPP 4.11 Mon 17:00 ZEU Lich

Confocal micro-Raman spectroscopy: A tool to discriminate calcified biominerals in land living crustaceans — •Sabine Hild and Andreas Ziegler — Central Facility for Electron Microscopy; University of Ulm, Albert-Einstein-Allee 11, 89081 Ulm

Land living crustaceans, like Porcellio scaber (Isopoda), have the ability to elaborate various types of calcified biominerals e.g. calcium phosphate, calcite, and amorphous calcium carbonate (ACC). Crystalline minerals are located mainly in the mineralized exoskeleton (cuticle) of the animals. Amorphous minerals, like ACC, which is thought to be a precurser for crystalline CaCO3, occur primarily in transient reservoirs for calcium. To get information about the morphology of the inorganic phase and their spatial distribution imaging techniques like REM and SFM are suitable. However, the various modifications of the biominerals cannot be discriminated by these techniques. Scanning confocal *-Raman spectroscopy (SCRS) enables to investigate the chemical composition of materials with a high resolution. In this study Raman spectral imaging in combination with SFM have been used to characterize and localize amorphous and crystalline minerals in a calcified tissue of P. scaber. First SCRS experiments reveal both crystalline and amorphous CaCO3 within the cuticle. Our model organism develops also transient reservoirs, which are fully X-ray amorphous. The reservoirs contain only ACC embedded in an organic matrix, which is proposed to stabilize the ACC. Additionally detected phosphate derivates suggests that they may also influence the stabilization of amorphous calcium carbonate.

CPP 4.12 Mon 17:15 ZEU Lich

Nanometer-sized temperature-sensors using the upconversion signal in single colloids of Lathanide-doped $NaYF_4$ — •Stefan Schietinger[1], Björn Lauritzen[1], Stephan Heer[2], Hans-Ulrich Güdel[2], and Oliver Benson[1] — [1]AG Nano-Optik, Institut für Physik, Humboldt-Universität zu Berlin, Hausvogteiplatz 5-7, 10117 Berlin (Germany) — [2]Department of Chemistry and Biochemistry, University of Bern, Freiestrasse 3, CH-3000 Bern 9 (Switzerland)

Because of low absorption of biological tissue in the near-infrared (NIR), luminescent biolabels based on two-photon absorption processes in this spectral region are of great interest. $NaYF_4$ colloids doped with Yb^{3+}/Er^{3+} with a size of 10-30 nm can be excited by photon upconversion. The excitation is based on sequential absorption of one photon by the Yb^{3+} ions followed by the absorption of a second photon and the energy transfer to Er^{3+} ions emitting the upconversion signal. Due to the contribution of a real metastable level in the Yb^{3+}, excitation rates are much higher than in two-photon absorption processes using a "virtual" quantum mechanical state[1].
Two states emitting around 540 nm are coupled thermally and therefore their intensity ratio is $\propto \exp(-\Delta E/k_B T)$. We were able to measure the temperature dependence in the biological interesting region around

300 K of a single nanocrystal. We therefore suggest not only the use of these colloids as stable and high efficient biomarkers, but also their employment as local temperature-sensors on a nanometer scale.

1 W. Krämer *et al.*, Chem. Mater. 16, 1244, (2004)

CPP 4.13 Mon 17:30 ZEU Lich

Blowing DNA bubbles — •Nikolai Severin[1], Wei Zhuang[1], Christof Ecker[1], Alexei A. Kalachev[2], Igor M. Sokolov[1], and Jürgen P. Rabe[1] — [1]Department of Physics, Humboldt University Berlin, Newtonstr. 15, D-12489 Berlin, Germany — [2]Plasmachem GmbH, Rudower Chaussee 29, D-12489 Berlin, Germany

Liquids at the interface with a solid differ from their bulk state since they order in molecular layers parallel to the substrate surface. With a surface forces apparatus, the layers can be squeezed out one by one at increasing normal forces, indicating solid-like behavior in the direction normal to the substrate. Similar layered behavior is observed for free liquid thin films on a surface. Computer simulations, measurements of shear forces and tracer dye diffusion, however, indicate liquid-like behavior parallel to the substrate. Therefore, we regard an ultra-thin layer of a liquid on a solid substrate as a quasi 2D-liquid. Here we demonstrate that such a layer may be used to transmit forces isotropically across a surface. A scanning force microscope (SFM) tip was employed to generate a 2D-pressure, which then exerts a force on a single macromolecule embedded in the 2D-liquid. In particular, supercoiled ds-DNA has been unraveled, moved, stretched, overstretched to 2.0 times its B-form length and then torn apart at (1.1 +/- 0.2) nN.