

CPP 11: Poster: New Instruments and Methods

Time: Monday 17:30–19:30

Location: P2

CPP 11.1 Mon 17:30 P2

Acoustical Characterization of PVDF-Transducers for Experiments on Musical Instruments — ●MARCEL KAPPEL, GUNNAR GIDION, and REIMUND GERHARD — University of Potsdam, Institute of Physics and Astronomy

Polyvinylidene fluoride (PVDF) has long been known for its flexibility in applications, which is based on its robustness and variable sensor geometry. Since the acoustical impedance is similar to that of materials like plastic and wood, it can be used for non-invasive investigations of vibrating systems for instance in musical instruments. Measurements of the piezoelectric coefficient of the PVDF-sensor were carried out with respect to important parameters such as static pressure, amplitude of the dynamical force, and long term stability. In addition, we determined the frequency response of the piezoelectric polymer as sensor and actuator. Thus, we are capable of using these transducers on two different musical instruments and compare the results with the airborne sound recorded simultaneously by a condenser microphone. These measurements can hold as a showcase for the application of PVDF-sensors in vibrating systems and material science in general.

CPP 11.2 Mon 17:30 P2

Calibration and application of novel thermal cantilever in the field of polymer science — ●THOMAS FISCHINGER and SABINE HILD — Department of Polymer Science, Johannes Kepler University Linz, Altenbergerstrasse 69, 4040 Linz, Austria

To satisfy the demands of designing polymers for advanced applications more detailed information about physical and chemical properties on the microscopic scale are required. Scanning probe microscopy has shown to be an proper tool. The development of a new generation of heatable cantilevers enhances prospects also into the nano-thermal characterization. A central point in the work with such probes is an appropriate and accurate temperature calibration of the probe. In this research the influences of sample preparation and experimental set-up on the calibration have been investigated. To minimized external influences the standard method is compared to the independent calibration of tip temperature using the Raman peak location method. With our basic experimental set-up, conventional Local Thermal Analysis in an unprecedented range of scale is available, which allows the detection of transition points such as melting point and softening point, possible. However, the determination of the glass transition point has reached its limits here, and is therefore being replaced by a new technique. Dual AC Resonance Tracking is a combination of atomic force acoustic microscopy and band excitation, which enables the unambiguous detection of temperature-induced changes in the contact stiffness of the tip-surface junction.

CPP 11.3 Mon 17:30 P2

Capability of specular reflection-absorption IR spectroscopy for qualitative and quantitative analysis of organic films on metal substrates — ●GEORG MAYR^{1,2}, SABINE HILD², and MICHAELA SCHATZL¹ — ¹voestalpine Stahl GmbH Linz, voestalpine-Straße 3, 4020 Linz, Austria — ²Institute of Polymer Science, Altenbergerstraße 69, J.K. University, 4020 Linz, Austria

Specular IR-reflection-absorption spectroscopy is a convenient tool for analysis of thin films of weak absorbing materials applied to highly reflecting substrates, e.g. metals. It is readily applied for practical purposes such as the analysis of organic coatings on metal sheets. Reflection spectra exhibit peculiarities compared to transmission spectra, what becomes evident from the investigation of films with various thicknesses (2-20 micrometer). Total absorption is not observed due to surface reflection. Weak absorption bands could be evaluated quantitatively. At intense bands deviations from the transmission mode band shape occur. These special features of reflection spectra could be explained by the refractive index dispersion and the therewith connected change in reflectivity of the coating layer in the wavelength area of an absorption band combined with Lambert-Beer absorption within the organic film. Under consideration of the aforementioned effects the reflection technique is capable of quantitative investigations, e.g. evaluation of the amount of remaining reactive functions within thermoset coatings.

CPP 11.4 Mon 17:30 P2

Characterization of functionalized graphene — ●MICHAEL ENZELBERGER¹, JAN ENGLERT², VIATCHESLAV DREMOV¹, FRANK HAUKE², ANDREAS HIRSCH², and PAUL MÜLLER¹ — ¹Department of Physics and Interdisciplinary Center for Molecular Materials (ICMM), Universität Erlangen-Nürnberg, Germany — ²Department of Chemistry and Pharmacy and Institute of Advanced Materials and Processes (ZMP), Universität Erlangen-Nürnberg, Germany

Covalent functionalization of graphene is a challenging task. A possible method for characterizing the reaction product is Raman spectroscopy. However its lateral resolution is limited to about 500nm. In this talk we present our scanning probe approach to this problem. Using Kelvin probe microscopy and AFM point contact spectroscopy under ambient conditions we analyzed local work function, adhesion and Young's modulus with a lateral resolution of 30 - 50 nm. We were able to correlate the spatially integrated averages of these results with the Raman spectroscopy data.

CPP 11.5 Mon 17:30 P2

Modeling Photothermal Detection, 3 Ways to make it Quantitative — ●MARKUS SELMKE, MARCO BRAUN, and FRANK CICHOS — Universität Leipzig, Experimental Physik I, mona, Deutschland

Photothermal spectroscopy has been long used to quantitatively study the absorption of liquids and materials in numerous variants, such as photothermal beam deflection spectroscopy and thermal-lens spectroscopy. While recent developments have pushed the sensitivity of its microscopic counterpart up to the level of a single quantum dot or even a single molecule, the technique has lost its quantitative character due to the complexity of the signal creation in a microscopy volume. Thus a quantitative description is missing and even the simple signal shape in axial photothermal scans of single metal nanoparticles remains unclear. Here we present 3 different quantitative descriptions reproducing the photothermal detection phenomena and parameter-dependencies while providing a thorough understanding of the signal generation. Full ab-initio EM-field calculations as well as simple Ray-Optics and Diffraction treatments are shown to agree well among each other and establish the interpretation in terms of an intuitive thermal lens mechanism. Application to experiments enable the extraction of absorption cross-sections from the photothermal signal of single nanoparticles.

CPP 11.6 Mon 17:30 P2

Photothermal Detection: New Experimental Perspectives from a Quantitative Theory — ●MARCO BRAUN, MARKUS SELMKE, and FRANK CICHOS — Molecular Nanophotonics Group, University of Leipzig, Linnéstraße 5, D - 04103 Leipzig

Photothermal microscopy has been used successfully for the detection of non-fluorescent nano-objects as gold nanoparticles, quantum dots or carbon nanotubes. Recently, the sensitivity of the method was improved to detect even single molecules only by absorption of light. The technique nevertheless suffers from a lack of a fundamental quantitative description. Within our group, a new Mie-like ab-initio EM-scattering theory was developed, capable to predict the photothermal signal in a quantitative manner. Experimental data will be presented supporting this theory and showing that the technique is based on the formation of a thermal nano-lens. This allows to investigate the temperature fields induced by a single optically heated nanoparticle. The gained insight opens up the opportunity of new applications of photothermal detection beyond the imaging of absorbing nano-objects. The absorption cross-section σ_{abs} may be extracted directly from the photothermal signal. Moreover, the theory suggests the application of dual focus photothermal correlation spectroscopy to probe the dynamics of absorbing nanoparticles.

CPP 11.7 Mon 17:30 P2

Imaging Polymer Nanostructures with Scanning Transmission X-ray Spectro-Microscopy — ●BENJAMIN WATTS¹, CHRISTOPHER MCNEILL², and JÖRG RAABE¹ — ¹Swiss Light Source, Paul Scherrer Institut, 5232 Villigen, Switzerland. — ²Cavendish Laboratory, University of Cambridge, J J Thomson Ave, Cambridge CB3 0HE, United Kingdom.

Thin polymer films are of increasing technological importance in optoelectronic devices such as LEDs, lasers, FETs and solar cells. However, the morphology of such films is complex, often displaying three dimen-

sional composition structure or molecular alignment effects. The structure of the polymer film incorporated into a device can strongly affect its performance characteristics, e.g. via the connectedness between of polymer domains and to the device electrodes, or due to anisotropic material properties due to molecular alignment.

Scanning transmission x-ray spectro-microscopy (STXM) has been demonstrated to be an excellent tool for the study of polymer materials due to its high spatial resolution and strong contrast based on a variety of spectroscopic mechanisms. Recent work at the Pol-Lux STXM has demonstrated two new developments in the imaging of thin polymer films: simultaneous surface and bulk imaging via an additional channeltron TEY detector, and molecular orientation mapping via anisotropic near edge resonances.

CPP 11.8 Mon 17:30 P2

Time-resolved soft X-ray Absorption Spectroscopy of Solvated Coordination Chemistry Compounds on a Microjet — ●ALEXANDER KOTHE¹, KAI F. HODECK¹, CHRIS J. MILNE², FRANK VAN MOURIK³, KATHRIN M. LANGE¹, MAJED CHERGUI³, and EMAD F. AZIZ^{1,4} — ¹Helmholtz Zentrum Berlin — ²Ecole Polytechnique Federale de Lausanne — ³Paul Scherrer Institut — ⁴FU Berlin

Investigation of the spin cross-over of coordination chemistry compounds in solution permits the study of the fundamental intramolecular processes in the absence of intermolecular effects. Time-resolved soft x-ray absorption spectroscopy (XAS) gives the opportunity to investigate especially the electronic and spin structure of molecular states and their dynamics. On a sub-fs scale this was achieved by means of dark channel fluorescence yield (DCFY), where L-edge XAS on FeCl₃ revealed the upper limit of 1-1.5 fs for the electron transfer to the solvent. Moreover we currently establish a system of ps-laser pump and L-edge XAS as a probe at BESSY. For this experiment we use [FeII(bpy)₃]2+ dissolved in water. Light of 532 nm wavelength excites a metal-to-ligand-charge-transfer state, which leads to an ultrafast spin change from singlet to quintet. The latter has a lifetime of 650 ps. Using a 23 μm microjet, the setup allows to constantly renew or cycle the sample. Besides circumventing sample damage, this gives direct access to the sample in vacuum for high repetition rate fluorescence yield measurements. This experiment opens the door for investigations of the dynamics of the charge transfer in heme proteins and other biochemical systems in solutions under real conditions.

CPP 11.9 Mon 17:30 P2

Contrast enhancement in STXM micrographs based on radiation damage — ●ANDREAS SPÄTH¹, JÖRG RAABE², and RAINER H. FINK¹ — ¹Friedrich-Alexander Universität Erlangen-Nürnberg, Physikalische Chemie II and ICMM, Erlangen, Germany — ²Swiss Light Source (SLS), Paul Scherrer Institut, Villigen, Switzerland

Radiation damage is not avoidable during resonant absorption of highly-energetic intense ionizing radiation like soft x-rays. In order to quantify its effect in x-ray microspectroscopy it is crucial to evaluate material specific critical doses as upper irradiation limit to detect the chemical species of interest and not its decomposition products. In particular in soft matter specimens sample degradation is particularly important [1]. For some materials, however, radiation damage can enhance the contrast in x-ray micrographs as will be demonstrated for microtomed slices of insect eyes (*xanthopan morgani* *predicta*). We can demonstrate that radiation energy dependent contrast enhancement is observed due to selective mass loss in anatomically different regions. The studies are completed by dose dependent studies of ultrathin polymer films and polymer bilayers with specific emphasis on the resonant and non-resonant excitations. We will employ these effects to quantify the contrast enhancement in STXM micrographs. In-situ AFM studies at the NanoXAS instrument offers direct correlations with local thickness variations [2]. The work is funded by the BMBF, contract 05K10WEA.

[1] J. Wang et al. *J. Electron Spectrosc. Rel. Phenom.* 170, 2009, 25

[2] I. Schmid et al. *J. Phys. Conf. Ser.* 186, 2009, 012015

CPP 11.10 Mon 17:30 P2

Quantitative determination of network porosity and degree of pore filling for inorganic-organic hybrid structures — GUNAR KAUNE¹, MARTIN HAESE-SEILLER², REINHARD KAMPMANN², JEAN-FRANCOIS MOULIN², QI ZHONG¹, and ●PETER MÜLLER-BUSCHBAUM¹ — ¹TU München, Physik Department, LS Funkt. Mat., James-Frank-Str. 1, 85748 Garching, Germany — ²Institut für Werkstofforschung, Helmholtz Zentrum Geesthacht, 21502 Geesthacht, Germany

In hybrid solar cells an inorganic semiconductor such as titania is in contact with a photoactive and semiconducting polymer. To achieve a large interface between both, bicontinuous network structures are very promising. We investigate the infiltration of a titania network structure with poly(N-vinylcarbazole) (PVK). The titania films show a hierarchical pore structure with mesopores 52 nm in size and additional large macropores with a diameter of about 180 nm. With the new method time-of-flight grazing incidence small-angle neutron scattering (TOF-GISANS) the quantitative porosity and the degree of pore filling are determined for different infiltration approaches [1].

[1] Kaune et al; *J. Poly. Sci. Part B.* 48, 1628-1635 (2010)

CPP 11.11 Mon 17:30 P2

First microbeam GISAXS Experiments at the new MiNaXS beamline P03 at PETRA III — ADELIN BUFFET¹, MOTTAKIN ABUL KASHEM¹, GUNTARD BENECKE^{1,2}, RALPH DOEHRMANN¹, RAINER GEHRKE¹, GERD HERZOG¹, VOLKER KOERSTGENS³, JAN PERLICH¹, MONIKA RAWOLLE³, ANDRE ROTHKIRCH¹, MATTHIAS SCHWARTZKOPF¹, KAI STASSIG¹, PETER MUELLER-BUSCHBAUM³, and ●STEPHAN V. ROTH¹ — ¹Deutsches Elektronen-Synchrotron (DESY), Notkestr. 85, D-22607 Hamburg, Germany — ²MPIKG Golm, Abt. Biomat., Wissenschaftspark Potsdam-Golm, D-14424 Potsdam, Germany — ³TU München, Physik Department, LS Funkt. Mat., James-Frank-Str. 1, 85748 Garching, Germany

The new Micro- and Nanofocus small- and wide angle X-ray Scattering undulator beamline (MiNaXS) P03 at the high-brilliance third-generation synchrotron radiation source PETRA III combines in a unique way low divergence and a microfocus and additionally a nanofocus beam. Being commissioned in 2010, the microfocus end station is operational. Using a microfocus beam size of 22x12 μm², a SAXS resolution of >100nm has already been achieved. We prove its versatility by combining in-situ the microfocus with grazing incidence SAXS (GISAXS) and imaging ellipsometry [1]. We present first results on in-situ experiments, utilizing the high time- and spatial resolution for drop-casting experiments, and give an outlook on the novel possibilities for investigating in real time structure formation upto mesoscopic length scales. [1] Koerstgens et al., *Anal. Bioanal. Chem.* 396, 139 (2010)

CPP 11.12 Mon 17:30 P2

The new beamline BL-1 at DELTA — ●THORSTEN BRENNER, DANIELA LIETZ, MICHAEL PAULUS, ULF BERGES, CHRISTIAN STERNEMANN, and METIN TOLAN — Fakultät Physik/DELTA, TU Dortmund, 44221 Dortmund.

Deep x-ray lithography is a method to produce various microstructures with a height up to several millimeters. Structures with a lateral resolution up to 2 μm and a high aspect ratio of about 100 can be produced. A well-established process for production of microstructures using lithography is the LIGA process. A wafer covered with a photo sensitive material is exposed through a mask. This mask consists of an x-ray transparent material and an absorber material. The lateral shape of the absorber is transferred into the photoresist by shadow projection. After chemical development the exposed (pos.) or unexposed (neg. resist) material is removed. Afterwards, the micromechanical components can be produced by electroplating and molding.

In 2010 the construction of beamline BL-1 at DELTA has been finished. This beamline uses white radiation generated by a 1.5 T bending magnet (critical energy 2.2 keV). First lithography exposures of SU-8 covered silicon wafers at BL-1 were performed successfully. The exposure times have been between five and ten minutes yielding a deposited bottom dose of 15 to 25 J/cm³ (top to bottom dose ratio ≈ 5). SEM images of the developed wafers show high quality structures concerning the adhesion between resist and substrate, aspect ratio, and wall roughness. Systematic studies to find optimal exposure parameters are planned for 2011.

CPP 11.13 Mon 17:30 P2

A new set-up for fast reflectivity measurements at BL9 of DELTA — ●FLORIAN WIRKERT, JULIA NASE, MICHAEL PAULUS, CHRISTIAN STERNEMANN, SEBASTIAN TIEMEYER, and METIN TOLAN — Fakultät Physik/DELTA, TU Dortmund, 44221 Dortmund

We report on a new setup for fast reflectivity measurements at beamline 9 of DELTA. A PILATUS-100k detector with a sensitive area of 83.8x33.5 mm² and a high dynamic range allows to obtain a complete reflectivity on a single TIFF image. The main difference to classical reflectivity measurements is that the detector remains fixed at its position while the sample is tilted continuously from 0° to an angle θ_{max}.

As first test measurements we investigated the adsorption of the protein lysozyme at the solid-liquid interface between a silicon wafer and water. In a sequence of reflectivities taking 55 s each, we could demonstrate the temporal evolution of protein adsorption at a solid-liquid interface. Thus this set-up provides a strong tool for the investigation of time-dependent processes by x-ray reflectivity measurements.

CPP 11.14 Mon 17:30 P2

The small angle scattering beamline BW4 of HASYLAB: Recent developments and future perspectives — ●JAN PERLICH, JAN RUBECK, STEPHAN V. ROTH, and RAINER GEHRKE — HASYLAB-DESY, Notkestr. 85, D-22603 Hamburg (Germany)

The small angle scattering wiggler beamline BW4 of HASYLAB, Hamburg (Germany), is a dedicated materials science beamline. The main experimental techniques are transmission small angle X-ray scattering (SAXS) as well as grazing incidence small angle X-ray scattering (GISAXS) in reflection geometry. The implemented microfocus option at the BW4 instrument enables experiments where lateral scanning of sample materials is feasible. After major refurbishments of the beamline in the recent years, the focus is on expanding the experimental capabilities by the access to an extended scattering q-range, e.g. grazing incidence wide angle X-ray scattering (GIWAXS), and the introduction of necessary sample environments for the structural characterization of novel material systems at specific physical conditions. In addition, in-situ sample observations in transmission or reflection, i.e. dip-coating with GISAXS, are possible with a fast detector enabling the structural characterization of time-dependent processes. We present the novel experimental capabilities based on selected recent results and give an outlook on upcoming activities.

CPP 11.15 Mon 17:30 P2

Molecular orientation in soft matter thin films studied by resonant soft x-ray reflectivity — ●PETER REICHERT¹, MARKUS MEZGER¹, BLANDINE JÉRÔME², and JEFFREY B. KORTRIGHT² — ¹Max-Planck-Institut für Polymerforschung, Mainz — ²Materials Sciences Division, Lawrence Berkeley National Lab., USA

Polarization resolved Resonant Soft X-Ray Reflectivity is a novel technique to study depth profiles of molecular orientation in soft matter thin films with nanometer resolution. It combines the chemical sensitivity of Near-Edge X-ray Absorption Fine Structure spectroscopy to specific molecular bonds and their orientation with respect to the polarization of the incident beam with the precise depth profiling capability of X-ray reflectivity. We show polarization dependent reflectivity data from side chain liquid crystalline polymer thin films and their quantitative analysis using the Berreman formalism. Further examples on buried films and amorphous polymers where the orientation is induced by the free surface and the substrate demonstrate the capabilities of the technique.

CPP 11.16 Mon 17:30 P2

CIDNP study of cyclic peptides with sulfur containing amino acids — ●TALEA KÖCHLING¹, GERALD HÖRNER¹, ALEXANDRA YURKOVSKAYA², and HANS-MARTIN VIETH¹ — ¹Fachbereich Physik, Freie Universität Berlin, Arnimallee 14, 14195 Berlin — ²International Tomography Center of SB RAS, 630090, Institutskaya 3a, Novosibirsk, Russia

Combining NMR spectroscopy with the effect of spin hyperpolarisation opens new ways to get information about structure of radicals and their reaction pathways. A way for creating transient non-thermal signal amplitudes is chemically induced dynamic nuclear polarisation (CIDNP) resulting from the formation of a radical pair of a photo-excited dye molecule and a quencher, e.g. an amino acid residue. The dependence of CIDNP on the external magnetic field allows to determine magnetic resonance parameters (hyperfine coupling constants and g-factor) of elusive radicals. On the other hand from its time dependence with microsecond resolution it is possible to differentiate geminate and bulk processes, so that reaction pathways and rate constants can be extracted.

In the present study, CIDNP results for different peptides containing a combination of methionine, methylcysteine and glycine in aqueous solution at ambient temperature are obtained. By comparing these peptides that differ in their sulfur-backbone-distances and number of thioether units differences in radical structure and reaction kinetics are

shown and discussed. In particular, primary and secondary reaction steps are differentiated and rate constants are determined.

CPP 11.17 Mon 17:30 P2

Spin-Flip NMR Spectroscopy as a Tool for Structure Investigations — ●GÜNTER HEMPEL and MATTHIAS ROOS — Martin-Luther-Universität, Institut für Physik, Betty-Heimann-Straße 7, D-06120 Halle

We propose a NMR procedure which can give information about molecular structure also in disordered systems. This method is based on polarization exchange between neighbored ¹H nuclei. The exchange rate is proportional to the coupling constant which depends on length and orientation of the spin-spin connection vector with respect to the magnetic field.

Because of the particular structure of the effective Hamiltonian, the oscillations survive relatively long time which can lead to rather high accuracy of the determination of the frequency and subsequently of the structural parameters. Coarsely spoken, we burn holes into the proton polarization and follow the subsequent re-filling of the holes. In microsecond range the oscillatory behaviour mentioned above is observed, and at millisecond scale spin-diffusion processes dominate.

This method was applied to liquid-crystal compounds to study certain bond angles in the mesomorphic phases which are not accessible by scattering methods. Furthermore, we could estimate spin-diffusion constants in liquid crystals and polymeric materials.

CPP 11.18 Mon 17:30 P2

NMR Signal Averaging in 62 Tesla Pulsed Fields — ●BENNO MEIER¹, SEBASTIAN GREISER¹, JÜRGEN HAASE¹, THOMAS HERRMANNSDÖRFER², FREDERIK WOLFF-FABRIS², and JOACHIM WOSNITZA² — ¹University of Leipzig, Faculty of Physics and Earth Science, Linnéstrasse 5, 04103 Leipzig, Germany — ²Hochfeld-Magnetlabor Dresden, Forschungszentrum Dresden-Rossendorf, 01328 Dresden, Germany

Nuclear Magnetic Resonance (NMR) experiments at up to 62 Tesla in pulsed high magnetic fields at the Hochfeldlabor Dresden-Rossendorf are reported. The time dependence of the magnetic field is investigated by observing various free induction decays (FIDs) in the vicinity of the maximum of the pulse. By analyzing each FID's initial phase and its evolution with time the magnetic field's time dependence can be determined with high precision. Assuming a cubic dependence on time near the field maximum its confidence is found to be better than ± 0.03 ppm at low fields and ± 0.8 ppm near 62 Tesla. The obtained time dependence of the field can be used to demodulate and phase correct all FIDs so that they appear phase locked to each other. As a consequence signal averaging is possible. The increase in signal-to-noise is found to be close to that expected theoretically. This shows that the intrinsic time dependence of these pulsed field can be removed so that the NMR signals appear to be taken at rather stable static field. This opens up the possibility of performing precise shift measurements and signal averaging also of unknown, weak signals if a reference signal is measured during the same field pulse with a double-resonance probe.

CPP 11.19 Mon 17:30 P2

Universal Long-Time Behavior of Spin Echo — ●BENNO MEIER¹, JONAS KOHLRAUTZ¹, BORIS FINE², and JÜRGEN HAASE¹ — ¹University of Leipzig, Faculty of Physics and Earth Science, Linnéstrasse 5, 04103 Leipzig, Germany — ²University of Heidelberg, Institute for Theoretical Physics, Philosophenweg 19, 69120 Heidelberg, Germany

Nuclear magnetic resonance (NMR) is a versatile tool also for the investigation of many-body dynamics in quantum mechanics. Here we report on preliminary measurements of free induction and solid echo decays of ¹⁹F spins ($I = 1/2$) in an isolated cubic lattice (CaF₂). In agreement with a recently developed generic theoretical description we find that the long-time decay of both solid echo and free induction decay (FID) is described by an exponentially decaying oscillatory function where exponential decay rate and frequency of oscillations are the same for both the FID and the solid echo and do not depend on the initial state of the spin system at the end of the pulse sequence. This behaviour supports the notion of eigenvalues of time evolution operators in chaotic many-body quantum systems.