CPP 28: New Instruments and Methods

Time: Tuesday 14:00–16:00 Location: C 264

CPP 28.1 Tue 14:00 C 264

New spectroscopic approaches for periodic systems — •Sandra Luber — Universität Zürich, Institut für Chemie, Winterthurerstr. 190, 8057 Zürich, Schweiz

Knowledge about local properties is extremely helpful for the analysis of structures and interactions. Moreover, it is a valuable source of information for the characterisation of dynamic processes and facilitates the interpretation of experimental data. In case of vibrational spectroscopy, for example, it is desirable to determine the impact of certain atoms/molecules on the bands in the experimental spectra. This may be straight forward for small systems but becomes more and more complex for larger systems.

Calculations provide additional insight allowing the targeted study of specific structures. In this way, it is possible to quantify the contributions of solute and solvent molecules [1] or adsorbates on solids.

We present novel, computationally efficient methods for the calculation of properties for periodic systems such as liquids and solids. These are applied to calculate, among others, vibrational spectra via ab initio molecular dynamics [2,3].

References:

[1] S. Luber, J. Phys. Chem. A 117 (2013) 2760. [2] S. Luber, M. Iannuzzi, J. Hutter, J. Chem. Phys. 141 (2014) 094503. [3] S. Luber, submitted.

CPP 28.2 Tue 14:15 C 264

Combination of 3D-cross correlated light scattering with small-angle neutron scattering: PNIPAM microgel particles as model system — •Annegret Günther[1,2], Yvonne Hertle[1], Daniel Clemens[2], Johannes Bookhold[1], Fangfang Chu[2], Matthias Ballauff[2], and Thomas Hellweg[1] — [1]Universität Bielefeld, PCIII, Universitätsstr. 25, 33615 Bielefeld — [2]Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, EM-ISFM, Hahn-Meitner-Platz 1, 14109 Berlin

The combination of 3D-cross correlation light scattering (3D-LS) with small-angle neutron scattering (SANS) will enable researchers, particularly in the field of soft condensed matter, to perform simultaneously neutron and light scattering experiments on highly concentrated samples. In order to show the applicability of this new setup PNIPAM microgel particles were used as a model system. For this kind of 'smart material' it was shown [1], that these particles in solution are deformed at higher particle concentrations. Since conventional light scattering experiments have to be performed on diluted samples to assure that only single-scattered light is detected, this concentration effect could only be observed by SANS. 3D-LS suppress the multiple scattering by performing two simultaneously light scattering experiments. By combining the 3D-LS with the SANS instrument V16 at the Helmholtz-Zentrum in Berlin, it will be possible to study samples under exact the same conditions for both scattering experiments.

[1] M. Stieger, J. S. Pedersen, P. Lindner and W. Richtering, Langmuir 20, 7283 (2004).

CPP 28.3 Tue 14:30 C 264

Non-destructive methods to determine the degree of crosslinking in polymeric materials — Gabriele Eder and •Volker Uhl — Austrian Research Institute for Chemistry and Technology (OFI), Vienna, Austria

The degree of crosslinking of many polymeric materials has a high impact on some technically important physical properties like hardness, elasticity, ductility or solubility. The main task of the work presented is to show that several quick and non-destructive characterization methods suited to determine the degree of crosslinking of polymeric component parts can be directly used to derive technically relevant physical or mechanical parameters. This can be achieved when a direct correlation between the measured and the physical properties of interest exist. As example, two series of variably cross-linked HDPE components were chosen, a material widely used for tubings and pipes, and investigated with various non-destructive methods. NIR-spectroscopy, confocal Raman spectroscopy and scanning acoustic microscopy proved to yield measured quantities directly correlated to the dose of irradiation used to crosslink the polymer. The results of these methods were validated against crosslinking data determined with conventional chemical analysis based on the solubility of the polymer. Furthermore, the degree of crosslinking was put into relation to characteristic mechanical parameters like Young's modulus and tensile strength, which were determined by mechanical testing.

CPP 28.4 Tue 14:45 C 264

Fiber enhanced Raman sensing of pharmaceutical drugs — •Torsten Frosch[1,2], Di Yan[1], and Juergen Popp[1,2] — [1]Leibniz Institute of Photonic Technology — [2]Institute of Physical Chemistry, Friedrich Schiller University Jena

Raman spectroscopy provides excellent molecular sensitivity for the analysis of pharmaceutical drugs and can also be applied in aqueous and biological environments. However the inelastic scattering process lacks high sensitivity. This drawback can be circumvented with help of innovative micro-structured hollow core fibers [1-3]. Such hollow fibers provide a miniaturized sample container for analyte flow and act at the same time as optical waveguide. Thus a very efficient interaction of the analyte molecules and the laser was achieved and the analytical sensitivity was highly improved for tracing molecules at very low concentrations [1]. Fiber enhanced Raman spectroscopy (FERS) was for the first time extended in the important UV spectral range1 and new concepts for optical waveguides in the deep UV were developed [2].

Acknowledgment: Funding of the research project by the Free State of Thuringia (Germany) and the European Union (EFRE) is highly acknowledged (FKZ: 2012 FGR 0013).

References: [1] Frosch, T.; Yan, D.; Popp, J. Anal Chem 2013, 85, 6264-6271. [2] Hartung, A.; Kobelke, J.; Schwuchow, A.; Wondraczek, K.; Bierlich, J.; Popp, J.; Frosch, T.; Schmidt, M. A. Optics Express 2014, 22, 19131. [3] Hanf, S.; Keiner, R.; Yan, D.; Popp, J.; Frosch, T. Anal Chem 2014, 86, 5278-5285.

CPP 28.5 Tue 15:00 C 264

Analysis of vibrating microstructures using dynamic scanning electron microscopy (DySEM) — •Maria Schröter[1], Martin Ritter[2], Matthias Holschneider[3], and Heinz Sturm[1] — [1]BAM 6.9, Federal Inst. Materials Research, Berlin — [2]Electron Microscopy, TU Hamburg-Harburg — [3]Inst. Mathematics, Univ. Potsdam

The term "DySEM" (Dynamic Scanning Electron Microscopy) denotes an experimental procedure for measuring the vibrational dynamics of a microscale oscillator using a scanning electron beam. The DySEM technique enables the direct observation of freely vibrating structures, including several modes in the normal and torsional direction as well as their higher harmonics. Additionally, the DySEM images contain characteristic amplitude-dependent image features.

Thus, this method is a tool of modal analysis of microscale structure in oscillation, which is frequently used in micro- and nanoelectromechanical systems (MEMS and NEMS) and where an optimization of the design parameters often only can be achieved by imaging the vibration (1). The advantage of DySEM technique is the ability to distinguish between artefacts based on the imaging process and features which carry relevant information (i.e. nonlinear mechanical behavior of the micro-oscillator, especially in tapping mode microscopy of soft matter). Our talk concentrates on experimental issues, the underlying theory of the experimental results is shown in a corresponding poster.

(1) Schröter; Dissertation TU Berlin (2014)

CPP 28.6 Tue 15:15 C 264

Characterization of deformation and fracture behaviour of semi-crystalline polymers by thermography with high lateral and time resolution — •Schneider Konrad[1] and Stein Marcus[2] — [1]Leibniz-Institut für Polymerforschung Dresden, Dresden, Germany — [2]Technische Universität Dresden, Fakultät Maschinenwesen, Institut für Verarbeitungsmaschinen und Mobile Arbeitsmaschinen, Germany

Plastic deformation during yielding and stretching as well as fracture propagation in semi-crystalline polymers dissipates a considerable amount of energy. This leads to a local temperature increase, connected with a change of local molecular mobility. By infrared thermography with high lateral and time resolution these changes can be quantified. With measurements on Poly(ethylene terephthalate) (PET) as well as Polycarbonate (PC) it will be demonstrated, how locally the glass transition temperature can be exceeded and how the deformation process in detail is affected by the really appeared tem-

perature. Also the appearance of cavitation will be mainly affected by the thermal fields in the necking zone. Finally the strongly located energy dissipation during fracture will be presented and discussed in detail. This discussion shall throw a new light on the relevant molecular rearranging processes.

CPP 28.7 Tue 15:30 C 264

Resolution Enhancement For Low-Temperature Scanning Microscopy By Cryostat Immersion Imaging — •Michael Metzger[1], Alexander Konrad[1], Alfred J. Meixner[1], and Marc Brecht[2] — [1]Institute of Physical and Theoretical Chemistry, University of Tuebingen, Germany — [2]Zurich University of Applied Science, Institute of Applied Mathematics and Physics, Winterthur, Switzerland

One convenient way to increase the resolution of e.g. fluorescence images is realized by confocal microscopy with an objective of high numerical aperture and immersion oil. The combination of immersion fluids with high-performance objectives is however exceedingly problematic under low temperature conditions. A new construction of a scanning stage and a well-chosen immersion fluid enables us to immerse an objective together with the sample positioned inside a cryostat at cryogenic temperatures. Heating the sample chamber over the melting point of an appropriate chosen immersion fluid (e.g. 1-propanol) allows us to move the objective into the melted immersion droplet and thereby to increase the refractive index between the objective lens and the sample. We recorded confocal fluorescence images of quantum dots at 160 K with a high NA objective. By determining the point spread function of imaged single quantum dots the effective numerical aperture was appointed to be larger than unity (1.08). The presented method provides new opportunities e.g. for studies on biological systems like vitrified cells at low temperature and is also of relevance for correlative light and electron cryo microscopy.

CPP 28.8 Tue 15:45 C 264

Stable Metallic Silver Nanostructures by Atomic Vapour Deposition on a Volatile Liquid Jet — Michael McNally, Gediminas Galinis, Oliver Youle, Ruth Chantry, and •Klaus von Haeften — Department of Physics and Astronomy, University Leicester, Leicester, LE1 7RH, UK

Synthesis of nanoparticles in liquids is a diverse field covering a range of powerful empirical methods to produce a variety of shapes, structures and compounds. The majority of methods are based on chemical reduction, inherently restricting the choice of starting materials and stabilisers. We show that by using a liquid micro jet it is possible to perform physical vapour deposition directly into a high vapour pressure liquid, producing a phase of intrinsically stable metallic silver nanoparticles. Silver nanoparticles produced by deposition directly into ethanol exhibit a plasmon resonance in the optical absorption spectrum which is unchanged over more than a year, indicative of metallic behaviour and exceptionally stability, without additives. TEM imaging show evenly dispersed spherical particles with a log-normal distribution of sizes peaking at 2.5 nm. Our observations suggest that atomic silver arrives at the liquid surface, becomes trapped, nucleates and grows into nanoparticles by subsequent collisions with silver atoms and ions. The particles grow step by step, or stabilise. This process occurs within the transit time of the liquid jet of 100 ms. Our method enable the investigation of metal vapour-solvent interactions of a practically unlimited range of metal-solvent combinations, the elucidation of the role of stabilisers and the production novel nanoparticles.