CPP 7: POSTER: INTERNAL SYMPOSIUM Scattering Experiments

Time: Monday 16:00–18:00 Location: Poster B

CPP 7.1 Mon 16:00 Poster B

Electric field effects on alignment of lamellar structures in diblock copolymer thin films studied by neutron scattering — •Xiuli Jiang[1], Thomas Gutberlet[2], Mukul Gupta[2], Thomas Geue[2], and Thomas Thurn-Albrecht[1] — [1]Department of Physics, Martin-Luther-University, Halle, Germany — [2]Laboratory for Neutron Scattering, ETH Zürich & PSI, Villigen, Switzerland

Self-assembled block copolymers have attracted much attention in recent years for their potential use as nanostructure templates. In this context, it is of interest to control the order and orientation of block copolymer thin films by means of external electric fields. In order to gain additional understanding of the alignment process, we studied the effect of a weak electric field on a lamellar microphase structure oriented perpendicular to the electric field by neutron diffuse scattering in reflection geometry. It has been predicted that the electric field induces periodic structural undulations which lead to a disruption of the original structure and facilitate reorientation to set in. In our experiments on lamellar PS-PMMA block copolymers these field-induced undulations were not observed. Rather, already after preparation the films displayed a diffuse intensity interpreted as corresponding to a mosaic structure of domains with a typical correlation length of about 1-2 μm. This diffuse intensity increases after the application of the field, even if the field is too weak to induce alignment. The lateral length scale of the mosaicity remains unaffected by the electric field.

CPP 7.2 Mon 16:00 Poster B

Extending the possibilities of a Kratky-Compact-Camera by use of focussing multilayer X-ray optics — •Thomas Henze, Klaus Schröter, and Thomas Thurn-Albrecht — Martin-Luther-Universität Halle-Wittenberg, Institut für Physik, Polymerphysik

The use of focussing multilayer x-ray optics in laboratory x-ray equipment offers the potential of a substantial gain in primary beam intensity without a significant loss of resolution. We present the result of a refurbishment of Kratky-Compact-Camera, a classical setup for small angle x-ray scattering on isotropic samples, with an elliptically bent focussing multilayer. The advantages of the Kratky collimation system are ease of alignment, high intensity and low background. A further gain in intensity is highly desirable for time dependent experiments as well as for measurements of weakly scattering samples. The performance of the revised setup is analyzed quantitatively by comparing intensity and full width at half maximum of the primary beam, as well as the minimal accessible scattering vector with the corresponding parameters of the simple setup without optics. A gain in intensity of a factor of 2 up to 10 is achieved, depending on the details of the alignment. In addition the multilayer produces a monochromatic beam. First measurements on exemplary polymer systems are shown.

CPP 7.3 Mon 16:00 Poster B

Structural investigation of casein micelles in thin films — •E. Metwalli[1], R. Gebhardt[1], A. Tolkach[2], J.-F. Moulin[1], V. Körstgens[1], S.V. Roth[3], R. Cubitt[4], and P. Müller-Buschbaum[1] — [1]Physikdepartment E13, TU München, 85747 Garching. — [2]Chair for Food Process Engineering and Dairy Technology, TU München, Weihenstephan — [3]HASYLAB at DESY, Hamburg — [4]Institut Laue-Langevin, Grenoble, France

Casein micellar films on solid supports have interesting applications in labeling of glass containers and adhesion technology. Gaining structural information on these films will help to optimize the appropriate physical properties for the desired application. Casein micelles thin films were applied from aqueous solutions on pre-cleaned glass slides by spin coating technique. The effect of transglutaminase enzyme concentrations on the thin film structures was investigated using atomic force microscopy (AFM) and grazing incidence small-angle X-ray scattering (GISAXS). The AFM images can only give information on the top surface structure with low statistical significance. In contrary, GISAXS provides information on the in-plane structures of the casein thin film with much higher statistical significance. The GISAXS data on the thin films were compared with those obtained by dynamic light scattering from the solution phase. The swelling behavior of some selected casein films in deuterated water vapor was also investigated using grazing incidence small-angle neutron scattering (GISANS). The results were discussed and compared with various proposed models on the casein micelle structures.

CPP 7.4 Mon 16:00 Poster B

Dielectric and neutron spectroscopy on nano-confined liquid crystals — Stefan Frunza[1], Ligia Frunza[1], •Andreas Schoenhals[2], Maria Mayorova[3], Reiner Zorn[3], and Bernhard Frick[4] — [1]National Institute of Materials Physics, R-76900-Bucharest-Magurele — [2]Federal Institute for Materials Research and Testing (BAM), — [3]Research Center Jülich, Institute for Solid State Research, D-52425 Jülich — [4]Institut Max von Laue - Paul Langevin, B.P. 156, F-38042 Grenoble

The liquid crystals E7 and 8CB are confined to the nanopores of the molecular sieve Al-MCM-41 with a mean diameter of the pores of 2.5 nm. Dielectric spectroscopy shows that for the confined system one relaxation process is observed. Its characteristic relaxation time is much lower compared to that of the bulk. The temperature dependence of the relaxation time of this relaxation process shows some similarities to glassy dynamics. Moreover excess contributions to the vibrational densities of state (Boson-Peak) were found by neutron scattering using the time-of-flight spectrometer IN4 at the ILL in Grenoble. The Boson- Peak is a characteristic feature of glassy behaviour. In addition elastic scans are carried out at the backscattering spectrometer IN10 (ILL) These measurements show a signature for glass transition.

CPP 7.5 Mon 16:00 Poster B

Influence of spacer length and density on the vertical structures of supported membranes studied by neutron reflectivity — •Peter Seitz[1], Oliver Purrucker[2], Anton Förtig[3], Raimund Gleixner[4], Giovanna Fragneto[5], Rainer Jordan[3], and Motomu Tanaka[1,2] — [1]Physikalisch-Chemisches Institut, Universität Heidelberg, Germany — [2]Physik-Department E22, Technische Universität München, Germany — [3]Institut für Technische Chemie, Technische Universität München, Germany — [4]Max Planck Institute of Biochemistry, Martinsried, Germany — [5]Institut Laue-Langevin, Grenoble, France

We studied the structure of a new class of polymer-supported membranes, which are separated from the solid substrate via poly(2-methyl-2-oxazoline) spacers of defined length, functionalized with a surface coupling group and hydrophobic membrane anchors. The proximal leaflet was deposited via Langmuir-Blodgett transfer, followed by vesicle fusion to deposit the distal layer. Precise control of the polymer chain length and its lateral density enables the quantitative adjustment of the thickness and the viscosity of the polymer interlayer. Previously, we measured the membrane-substrate distance with fluorescence interference contrast microscopy (FLIC). To gain a deeper insight to the vertical structure of the membrane, we conducted specular neutron reflectivity experiments under a systematic variation of the spacer length and density, and calculated the static roughness and the volume fraction of water in the polymer interlayer.

CPP 7.6 Mon 16:00 Poster B

Formation of lateral structures in thin diblock copolymer films by vapor treatment — •Christine Papadakis[1], Peter Černoch[2], Charles Darko[1], Ezz Metwalli[1], Petr Štěpánek[2], Detlef-M. Smilgies[3], and Stephan V. Roth[4] — [1]Physikdepartment E13, TU München, James-Franck-Str. 1, 85747 Garching — [2]Inst. Macromolecular Chemistry, Prague, Czech Republic — [3]Cornell University, Ithaca NY, USA — [4]HASYLAB at DESY, Hamburg

Diblock copolymers in the melt spontanously self-organize into mesoscopically ordered structures. In order to understand their response to changes of the environment, in-situ and real-time methods are of great value. We have performed grazing-incidence small-angle X-ray scattering (GISAXS) measurements on thin films of poly(4-octylstyrene-*b*-butyl methacrylate) (OB) and poly(4-octylstyrene-*b*-methyl methacrylate) (OM) before and after vapor treatment with different solvents. In the bulk, OB is lamellar, whereas OM forms connected struts [1]. The solvent quality and selectivity were found to have a strong influence on the inner film structure. Solvents which are poor for one block and good for the other block preferentially lead to the formation of lateral structures. Time-resolved in-situ GISAXS measurements allowed us to follow the formation of lateral structures in the vapor, giving insight into the time scales and the mechanisms involved.

1. P. Černoch, P. Štěpánek et al., Eur. Polym. J., accepted.

CPP 7.7 Mon 16:00 Poster B

Recent developments at BW4 / HASYLAB — •A. Timmann, S.V. Roth, R. Gehrke, S. Hu, F.-U. Dill, M. Dommach, R. Döhrmann, and T. Schubert — HASYLAB at DESY, Notkestr. 85, D-22607 Hamburg, Germany

The beamline BW4 of HASYLAB, Hamburg (Germany), is a dedicated materials science beamline [1]. The main experimental techniques used are transmission small-angle x-ray scattering (TSAXS) and grazing-incidence SAXS (GISAXS). With the implementation of the microfocus option at BW4 scanning experiments have become possible [1]. After the major refurbishment of the beamline in 2004 and 2005, we focussed on upgrading and introducing necessary sample environments. This includes a new stretching cell allowing to draw film shaped samples to elongations up to 640mm with drawing velocities from 0.1 up to 1200 mm/min keeping the beam position on the sample fixed. Presently, several force sensors with a measuring range up to 500N are available now, which will be upgraded to 1kN. For GISAXS, a heating stage for polymeric samples has been commissioned. In-situ sample observation is now possible with a high-resolution video camera. Based on recent experimental results, we present the upgraded capabilities of BW4.

[1] S.V. Roth et al., Rev. Sci. Instr. 77, 085106 (2006)

CPP 7.8 Mon 16:00 Poster B

Structure and changes of thin block copolymer films during vapor treatment — •zhenyu di[1], christine papadakis[1], dorthe posselt[2], and detlef-m smilgies[3] — [1]Physikdepartment E13, TU München, 85747 Garching, Germany — [2]IMFUFA, Roskilde University, Dänemark — [3]CHESS, Cornell University, Ithaca NY, USA

The mesoscopic structures formed by diblock copolymers in thin film geometry are interesting for a variety of applications. However, self-assembly leads to domain structures with domain walls and defects. Vapor treatment has been shown to be an efficient way to increase the long-range order.

We have studied the structural changes in thin films of poly(styrene-b-butadiene) (PS-PB) diblock copolymers and their kinetics during vapor treatment. In this system, the initial lamellar orientation (parallel or perpendicular) can be controlled by the block copolymer molar mass [1]. We have investigated both initial orientations as well as the influence of the solvent selectivity and the film thickness. In-situ grazing-incidence small-angle x-ray scattering (GISAXS) is ideally suited because of its good time resolution (˜sec) [2]. We found that in addition to swelling of the films, lamellar reorientation takes place on the time scale of minutes. Complex processes and transient states were encountered. In some cases, improved long-range order was achieved.

[1] P. Busch, D. Posselt, D.-M. Smilgies, C.M. Papadakis et al. Macromolecules 36, 8717 (2003) and Macromolecules, in press.

[2] D.-M. Smilgies, P. Busch, C.M. Papadakis, D. Posselt, Synchr. Rad. News 15(5), p. 35 (2002).

CPP 7.9 Mon 16:00 Poster B

Thin films of diblock copolymers having one crystalline block — •charles Darko[1], ezzeldin Metwalli[1], ioan Botiz[2], günter Reiter[2], dag w Breiby[3], stephan v Roth[4], detlef-m Smilgies[5], and christine m Papadakis[1] — [1]Physikdepartment E13, TU München, James-Franck-Str. 1, D-85747 Garching — [2]Institut de Chimie de Surfaces et Interfaces, CNRS, Mulhouse, France — [3]Niels Bohr Institute, University of Copenhagen, Denmark — [4]Hasylab at DESY, Hamburg — [5]CHESS, Cornell University, Ithaca NY, USA

In thin films of semicrystalline diblock copolymers, the final structure formed depends on the competition between (i) the order-disorder transition of the diblock copolymer, (ii) the crystallization of the crystallisable block, and (iii) the vitrification of the amorphous block. We have studied lamellae-forming poly(styrene-b-ethylene oxide) diblock copolymers differing in molar mass. Combining grazing-incidence small and wide-angle X-ray scattering with high-resolution grazing-incidence X-ray diffraction, we obtain information on the inner structures on a large range of length scales and can compare them to the surface textures from optical and atomic-force microscopy. For the lowest molar mass sample, we find that the surface textures are different from the ones of poly(ethylene oxide) homopolymer films and that, depending on crystallization temperature, confined crystallization or breakout occur.

CPP 7.10 Mon 16:00 Poster B

The microfocus SAXS/WAXS beamline at PETRA III / DESY — •S.V. Roth, R. Döhrmann, H. Franz, R. Gehrke, U. Hahn, R. Röhlsberger, H. Schulte-Schrepping, N. Schwarz, and E. Weckert — HASYLAB at DESY, Notkestr. 85, D-22607 Hamburg, Germany

Among the first beamlines to be built at PETRA III is the microfocus small- and wide-angle x-ray scattering beamline μSAXS/WAXS. This beamline will exploit the excellent photon beam properties of the low emittance source PETRA III to provide micro- and nanofocused beams in dedicated end-stations with ultra-high intensity and resolution in real and reciprocal space. Targeting at scanning investigations at multiple length scales, the areas of research include in-situ experiments as well as brilliance demanding novel methods like microbeam grazing incidence small-angle x-ray scattering (μGISAXS) [1] and microbeam SAXS tomography [2]. The current layout foresees a large-offset double crystal monochromator and compound refractive on-axis optics for microfocusing. The projected beam sizes for routine user operation range from 110nm-40μm. The design of the beamline especially allows for combining ultra small-angle x-ray scattering (USAXS) with a microfocused beam (μUSAXS). We will present the present layout of the μSAXS/WAXS beamline introducing the projected capabilities of this new micro- and nanofocus scattering beamline at PETRA III.

[1] S.V. Roth et al., Appl. Phys. Lett. 88 (2006) 021910

[2] C.G. Schroer et al., Appl. Phys. Lett. 88 (2006) 164102

CPP 7.11 Mon 16:00 Poster B

High Pressure SAXS/WAXS using a Diamond Anvil Cell — •Ronald Gebhardt, Manfred Burghammer, Michael Hanfland, Mohamed Mezouar, and Christian Riekel — European Synchrotron Radiation Facility, B.P. 220, F-38043 Grenoble Cedex, France

The possibility of using a diamond anvil cell for SAXS/WAXS studies on small confined volumes has been explored at the ESRF ID13 beamline. As a demonstration experiment, single potato starch granules were pressurized up to 700 MPa at 20°C. In this pressure range a two-phase process was proposed (1,2). Scanning SAXS/WAXS experiments were performed with an about 1 micron beam. A Raman spectrometer was used to determine the pressure from the ruby fluorescence. The experimental setup is explained and first experimental results are shown. 1) Svensson E., Eliasson A. C. (1995) Carbohydrate Research, 26, 171-176 2) Rubens P., Heremans K. (2000) Biopolymers, 54, 524-530

CPP 7.12 Mon 16:00 Poster B

Spin-Echo Neutron Reflectivity on Diblock-Copolymer Films — •Max Nülle, Adrian Rühm, Marton Major, János Major, and Helmut Dosch — Max Planck Institut für Metallforschung, Heisenbergstr. 3, 70569 Stuttgart

SERGIS (Spin-Echo Resolved Grazing Incidence neutron Scattering) is a recently developed novel neutron scattering technique which we employ at the new neutron/X-ray reflectometer N-REX+ at FRM II. In contrast to conventional scattering methods, SERGIS measures the lateral structure and morphology of surfaces and thin film systems in real space. The spatial resolution is achieved by measuring the total polarization of the scattered beam, without the usual need to collimate the beam in the direction of interest. Therefore SERGIS combines a high neutron flux with the ability to characterize structures from the nanometer scale up into the micron range, and can thus yields novel information about both equilibrium and time-dependent phenomena on these length scales. As an example, we employ SERGIS to study morphologies produced by dewetting phenomena in thin polymer films. These experiments are complemented by detailed atomic force microscopy investigations. First results on thin diblock-copolymer films will be presented.

CPP 7.13 Mon 16:00 Poster B

Ellipsometric Light Scattering: The Spectroscopic Approach — •Arne Stark[1], Andreas Erbe[2], Klaus Tauer[1], and Reinhard Sigel[1] — [1]MPI of Colloids and Interfaces, D-14476 Golm — [2]Institute of Physics, Academia Sinica, Taiwan

We present first results obtained by Spectroscopic Ellipsometric Light Scattering (SELS). The discussion is complemented by an analysis of the general robustness of ELS against polydispersity effects.

Ellipsometric light scattering is based on Mie theory to analyze the scattering of polarized light by suspended particles in the colloidal range. Similar to standard ellipsometry this technique has an enhanced

sensitivity to interface effects, allowing to investigate e.g. (multiple) coatings on particles or adsorbed surface layers.

First experimental results obtained with a multi-wavelength apparatus are presented: augmenting the method to a spectroscopic technique extends the resolution considerably. The camera detector allows a simultaneous investigation of a range of scattering angles and speeds up the measurements. Light of different wavelengths is selected by interference filters from a mercury lamp. Thus, it is established experimentally that coherence of the light source is not required.

All ellipsometric methods yield ratios of eigenvalues of the optical system under investigation. Consequently ELS exhibits an averaging behavior that is distinctly different from normal static light scattering. It is shown theoretically and experimentally that information regarding structural details of the suspended particles is robustly preserved in the presence of moderate sample polydispersity.

CPP 7.14 Mon 16:00 Poster B

Polymer coated micro mechanical cantilever arrays — •Sebastian K. Nett[1,2], Gunnar Kircher[1], and Jochen S. Gutmann[1,2] — [1]Max Planck Institute for Polymer Research, Ackermannweg 10, D-55128 Mainz, Germany — [2]Institute for Physical Chemistry, Johannes Gutenberg University, Jakob-Welder-Weg 10, D-55099 Mainz

Functional layers can turn micro mechanical sensors consisting of an array of eight cantilevers into specific sensing systems for chemical or biological applications. We showed before that tethered polymers are robust and sensitive for analytes. To access individual cantilevers inkjet printing is a versatile approach. Using the automatic pipetting system, Nano-Plotter^{TM} (GeSiM) the solution for tethering is applied contact less in small droplets directly onto the cantilever. Small droplets tend to evaporate in a few seconds. On the other hand anchoring the molecules to the surface need some time due to kinetic and diffusion processes. Therefore room temperature Ionic Liquids which have a negligible vapour pressure are incorporated in the coating step. The small geometry of the cantilever (500 to 1000 μm length, 90 μm width, 1 to 5 μm of thickness) rises problems with the standard techniques for characterising the functional layer. To overcome these characterization problems, we used the μ-focus option at the GISAXS Line BW4 at the HASYLAB in Hamburg for detailed studies.

CPP 7.15 Mon 16:00 Poster B

Investigation of lubrication in natural joints by neutron reflectometry — •Thomas Kaltofen[1], Thomas Gutberlet[2], Maximilian Wolff[3], Roland Steitz[4], and Reiner Dahint[1] — [1]Angewandte Physikalische Chemie, Universität Heidelberg — [2]Paul-Scherrer-Institut, Villigen, Schweiz — [3]Experimentalphysik IV - Festkörperphysik, Universität Bochum — [4]Hahn-Meitner-Institut, Berlin

Despite their high medical relevance, the principles of lubrication in natural joints are still unclear. It is generally accepted, that the presence of hyaluronic acid (HA), the main component of the synovial liquid, plays an important role for the low friction observed. Furthermore, it is assumed that surface active lipids participate in the lubrication. Using a model system of lipid bilayers deposited on a polyelectrolyte (PE) cushion and in contact with HA solution, we started to investigate the effects of pressure and shear forces, as experienced by natural joints, on the internal structure of the SiO_2/PE/lipid/HA interface and the bulk HA solution by neutron reflectometry (NR), complemented by in situ ellipsometry and quartz crystal microbalance (QCM-D) measurements. Only on positively charged polyelectrolyte surfaces, the successful build-up of the model system could be demonstrated. By NR, the existence of an irreversibly absorbed, highly hydrated HA layer on top of the lipid membrane was proven. For shear rates above 2.5 min^{-1} a swelling of the HA layer has been observed. Pressure dependent studies are presently underway.

CPP 7.16 Mon 16:00 Poster B

Influence of degree of branching in LLDPE on its physical properties — •Stefan Fischer[1], Dieter Meinhardt[2], Bernhard Rieger[2], and Othmar Marti[1] — [1]Institute of Experimental Physics, University of Ulm, 89069 Ulm, Germany — [2]Institute of Inorganic Chemistry II, University of Ulm, 89069 Ulm, Germany

Novel metallocene catalysts allow the polymerization of linear low density polyethylene (LLDPE) with controlled molecular weight directly from ethylene without the need for (more expensive) higher alkanes [1]. The influence of the degree of branching and the molecular weight on the physical properties has been analyzed with several methods, including tensile tests, AFM (atomic force microscopy), DSC (differential scanning microscopy), WAXS (wide angle x-ray scattering) and SAXS (small angle x-ray scattering). The LLDPE samples have been compared with commercially available high and low density polyethylenes (HDPE and LDPE). The results will be presented.

[1] Dieter Meinhard, Marcus Wegner, Georgy Kipiani, Andrew Hearley, Peter Reuter, Stefan Fischer, Othmar Marti and Bernhard Rieger; JACS; submitted

CPP 7.17 Mon 16:00 Poster B

Modelling the structure of fillers in rubber and their small angle scattering — •Klaus Nusser, Gerald Johannes Schneider, and Dietmar Göritz — Universität Regensburg, Universitätsstr. 31, 93053 Regensburg

Silicas are technically important fillers for elastomers with respect to several applications. In such a filler-rubber system the mechanical properties strongly depend on the kind and the concentration of the filler. Despite being cruciable for the adjustment of high performance materials, the exact correlation between the morphology of the filler and the related reinforcement of the material is still unknown.

An up-to-date constitutive model suggests that isotropic silica primary particles form fractal clusters, which work as a basic unit for larger cluster structures. This notion was gained by the analysis of small angle X-ray scattering curves, which show regions of constant slope in a log-log plot. However, conclusions from scattering curves are not unique. In order to examine the assumptions made in constitutive models, numerical investigations were performed. Scattering structures were generated and their scattering curves were calculated by means of a computer. In particular, the origin of regions of constant slope in log-log depicted scattering curves was investigated.

CPP 7.18 Mon 16:00 Poster B

Side chain crystallization in soft and rigid confinement — •E. Hempel[1], H. Budde[2], M. Hahn[2], and M. Beiner[1] — [1]Martin-Luther-Universität Halle-Wittenberg, Institut für Physik, D-06099 Halle (Saale), Germany — [2]Fraunhofer Pilotanlagenzentrum für Polymersynthese und -verarbeitung, Value Park, Bau A74, D-06258 Schkopau, Germany

The side chain crystallization of poly(n-octadecyl methacrylate) [PODMA] under confinement is studied in a series of microphase-separated block copolymers containing PODMA cylinders with diameters in the range 10-20nm. The glass temperature of the matrix is systematically varied in the range 50...-30°C by choosing amorphous poly(n-alkyl methacrylates) with three to eight alkyl carbons per side chain as second component of the block copolymer. Since the crystallization temperature of the PODMA block is about 25°C crystallization occurs either in a glassy or in a highly viscous environment. The influence of the matrix properties on crystallization kinetics and internal structure of the PODMA domains is investigated by DSC and X-ray scattering. A central question of this study is whether or not the block copolymer morphology is changing due to side chain crystallization in the PODMA domains under conditions where the matrix material is soft and chemically not too different from the crystallizable component.

CPP 7.19 Mon 16:00 Poster B

Reversible Tuning of Block Copolymer Domain Spacings via Electric Fields — •Kristin Schmidt[1], Heiko Schoberth[1], Helmut Hänsel[1], Thomas Weiss[2], Volker Urban[3], Alexander Böker[1], and Georg Krausch[1] — [1]Lehrstuhl für Physikalische Chemie II, Universität Bayreuth, D-95440 Bayreuth, Germany — [2]European Synchrotron Radiation Facility (ESRF), F-38043 Grenoble, France — [3]Oak Ridge National Lab (ORNL), Oak Ridge, TN 37831, USA

We investigate the influence of electric fields on the domain spacing of a lamellar forming diblock copolymer solution by time-resolved synchrotron small angle X-ray scattering. As a model system we use a polystyrene-*b*-polyisoprene block copolymer dissolved in toluene or tetrahydrofurane. We find a significant dependence of the characteristic spacing on the electric field strength. For lamellae aligned parallel to the electric field direction we observe a decreasing lamellar distance with increasing field strength, while for perpendicularly orientated lamellae the domain size is increasing. We investigate the influence of the electric field strength, the degree of phase separation, molecular weight, composition and the polarity of the solvent. Furthermore we monitor the relaxation kinetics and show the revesibility of the changes in domain spacing on application and turn off of the electric field.

CPP 7.20 Mon 16:00 Poster B

Scaling behavior of the reorientation kinetics and changes in the phase behavior of block copolymers exposed to electric fields — •Heiko G. Schoberth[1], Kristin Schmidt[1], Thomas M. Weiss[2], Agur Sevink[3], Andrei V. Zvelindovsky[4], Alexander Böker[1], and Georg Krausch[1] — [1]Lehrstuhl für Physikalische Chemie II, Universität Bayreuth, D-95440 Bayreuth, Germany — [2]European Synchrotron Radiation Facility (ESRF), F-38043 Grenoble, France — [3]Leiden Institute of Chemistry, Universiteit Leiden, The Netherlands — [4]Department of Physics, Astronomy & Mathematics, University of Central Lancashire, Preston PR1 2HE, United Kingdom

We have followed the reorientation kinetics of various block copolymer solutions exposed to an external electric DC field by time-resolved synchrotron small-angle X-ray scattering (SAXS). The characteristic time constants follow a power law indicating that the reorientation is driven by a decrease in electrostatic energy. Moreover, the observed exponent suggests an activated process in line with the expectations for a nucleation and growth process. When properly scaled, the data collapse onto a single master curve spanning several orders of magnitude both in reduced time and in reduced energy. The power law dependence of the rate of reorientation derived from computer simulations based on dynamic density functional theory (MesoDyn) agrees well with the experimental observations. In addition, we observe an influence of the electric field on the order disorder temperature and phase separation in our block copolymer solutions. For further investigations we study this effect by birefringence and SAXS.

CPP 7.21 Mon 16:00 Poster B

Characterisation of structural changes during deformation and relaxation of semi-crystalline polymers by SAXS — •Konrad Schneider[1] and Peter Bösecke[2] — [1]Leibniz-Institut für Polymerforschung Dresden, Hohe Str. 6, 01069 Dresden — [2]European Synchrotron Radiation Facility (ESRF), BP 220, F38043 Grenoble, France

Time resolved small angle x-ray scattering using synchrotron radiation allows to follow up structural changes during deformation and relaxation of semi-crystalline polymers. On the example of polypropylene as well as high-density polyethylene copolymers the changes of the structure in the crystalline and in the amorphous domains were followed during the three characteristic stages of the load-displacement curves: The elastic stage and the plastic range composed of the stage of the lowering load in the force-displacement-curve (yielding) and the strain hardening. It is found that most rigorous changes are observed during yielding, whereas changes on a scale of second during relaxation are more pronounced than during the loading steps.

CPP 7.22 Mon 16:00 Poster B

Upgrades of two SANS diffractometers at the Munich reactor FRM-II — •Henrich Frielinghaus, Aurel Radulescu, Peter Busch, Vitaly Pipich, Alexander Ioffe, Dietmar Schwahn, and Dieter Richter — Forschungszentrum Jülich GmbH, Jülich Centre of Neutron Science, D-52425 Jülich

Political decisions made the Jülich reactor FRJ-2 shut down, and the high performance instruments are moved to Munich now. The two SANS diffractometers are transferred, and refurbished in a second step in order to meet the world leading machines. Neutron polarization and polarization analysis will be possible to study magnetic samples. Supermirrors and ^{3}He filters will be installed. Aspherical neutron MgF_2 lenses aim at two goals: 10 to 20 times higher intensity and a resolution of $\Delta Q = 10^{-4}$Å. The lenses will be cooled to minimize the thermal diffuse scattering. A high resolution detector ($\Delta x = 0.5$mm) allows to collect the high resolution scattering patterns. To operate with high resolution at finite Q a chopper placed behind the selector will reduce the wavelength uncertainty to 1% and better by a TOF analysis. The newly developed TiSANE mode will be possible: The neutron pulses will be triggered by a periodic external field at the sample in concert with the projected time-dependent scattering collected by the detector. We expect to resolve kinematic processes in the range of 2μs to 5ms and above. In this mode the Q-resolution typically is not improved. The GISANS option allows to study the lateral structure of thin films in the range of 10 to 1000Å with a free depth resolution. Here we aim at soft condensed matter and magnetic films.

CPP 7.23 Mon 16:00 Poster B

Focusing-mirror ultra-small-angle neutron scattering (USANS) instrument of Research Centre Jülich at FRM-2 Reactor — Emmanuel Kentzinger[1], •Aurel Radulescu[1,2], Alexander Ioffe[1,2], Peter-Paul Stronciwilk[1,2], Jörg Stellbrink[1], Dietmar Schwahn[1], Thomas Brückel[1,2], and Dieter Richter[1,2] — [1]Institute for Solid State Research, Research Centre Jülich, 52425 Jülich, Germany — [2]Jülich Centre for Neutron Sciences (JCNS), Research Centre Jülich, 52425 Jülich, Germany

Biological, colloidal and macromolecular samples presenting characteristic length scales from nanometer up to micron-scale are usually investigated by combining USANS at double-crystal diffractometers (DCD) and SANS at conventional pinhole cameras. In principle, the Q-range of both classes of instruments overlaps but the required instrumental settings push both techniques to their limits, mainly due to signal-to-noise level and the reduced flux at sample position. The KWS3 USANS instrument of Research Centre Jülich is the worldwide unique instrument running on the principle of a one-to-one image of an entrance aperture on a two-dimensional position sensitive detector by neutron reflection from a double-focusing toroidal mirror. The instrument is just relocated at the FRM-2 reactor in Garching where it will permit to perform SANS studies within a Q-range between $4x10^{-5}$ and 10^{-3} Å^{-1} with considerable advantages over the DCD and conventional SANS instruments. The working principle and recent use of KWS3 in investigations of multiscale aggregates formed by poly(ethylene-butene) random copolymers in solution are reported.

CPP 7.24 Mon 16:00 Poster B

Microbeam GISAXS investigation of sol-gel templated nanocomposite films — •J. Perlich[1], M. Memesa[2], J.S. Gutmann[2,3], S.V. Roth[4], and P. Müller-Buschbaum[1] — [1]TU München, Physik-Department LS E13, James-Franck-Str. 1, D-85748 Garching (Germany) — [2]Max-Planck Institute for Polymer Research, Ackermannweg 10, D-55128 Mainz (Germany) — [3]Institute for Physical Chemistry, Johannes Gutenberg University, Jakob-Welder-Weg 10, D-55099 Mainz (Germany) — [4]HASYLAB at DESY, Notkestr. 85, D-22603 Hamburg (Germany)

We focus on the creation of nanostructured TiO2 thin films, which are of great interest for many applications, due to their electrical performance. The performance is strongly dependent on the morphology of the nanocomposite films. For the preparation of the TiO2 nanocomposite films we combine the amphilic diblock-copolymer PS-b-PEO with an inorganic sol-gel chemistry. Under these conditions a so-called good-poor-solvent pair induced phase separation leads to the formation of the nanostructures by film preparation via spin-coating. The different morphologies can be controlled by the solvent concentration. In order to obtain crystalline TiO2 films as the final step calcination is conducted at higher temperature in air. For the investigation of the morphologies of the sol-gel templated nanocomposite films microbeam grazing incidence small angle x-ray scattering (μGISAXS) is performed at the synchrotron beamline BW4 of the DESY HASYLAB. The investigation is complemented by surface and thin film sensitive probes. We acknowledge financial support by the project MU 1487/5-1.

CPP 7.25 Mon 16:00 Poster B

The new Jülich Neutron Spin Echo Spectrometer J-NSE at the FRM-II — •Olaf Holderer[1,2], Michael Monkenbusch[1,2], Reinhard Schätzler[1,2], and Dieter Richter[1,2] — [1]Jülich Centre for Neutron Science (JCNS), Forschungszentrum Jülich GmbH, D-52425 Jülich, Germany — [2]Institut für Festkörperforschung, Forschungszentrum Jülich GmbH, D-52425 Jülich, Germany

Neutron spin echo (NSE) spectroscopy is a well-suited method for studying the dynamics of soft matter systems such as glasses, polymers and complex liquids and paramagnetic properties of e.g. spin glasses. The Jülich NSE spectrometer has been in operation at the Jülich research reactor FRJ-2 since 1996. It has been transferred now to the new research reactor FRM-II of the TU München, where it is in the comissioning phase and will continue with user service this year. The J-NSE will operate at an neutron guide end position, giving access to the wavelength band of about 4.5 to 16 Å. New correction coils have been designed and manufactured, allowing to use higher currents in the main precession coils.

A larger neutron guide exit of 60x60 mm, the higher neutron flux and the better correction elements push the performance of the instrument to fouriertimes λ=1 ps to about 350 ns, with a q-range of q=0.02 - 1.5 1/Å.

CPP 7.26 Mon 16:00 Poster B

GISAXS studies on titania ultrathin films with different morphologies — yajun cheng[1], markus wolkenhauer[1], gina bumbu[1], stephan roth[3], jochen gutmann[1,2], and •yajun cheng[1] — [1]Max

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Ultrathin TiO2 films showing rich morphologies are achieved by using sol-gel chemistry coupled with an amphiphilic polystyrene-block-poly (ethylene oxide) (PS-b-PEO) diblock copolymer as a structure-directing agent. The block copolymer undergoes a good-poor-solvent pair induced phase separation in a mixed solution of 1,4-dioxane, concentrated hydrochloric acid (HCl) and Titanium tetraisopropoxide (TTIP). By adjusting the weight fractions of 1,4-dioxane, HCl, and TTIP, inorganic-block-copolymer composite films containing a variety of different morphologies are obtained. Through calcination the amorphous Titania composite films can be converted to crystalline anatase phase. Based on the local characterization of AFM and SEM on the various different morphologies of the film. Grazing incidence small angle x-ray scattering (GISAXS) is further conducted to study the lateral structure of the film over large length scale. As a complementary method, x-ray reflectivity experiment is made to investigate the electron density gradient normal to the substrate. As a result, the combination of local imaging characterization and average x-ray scattering experiment provide a deep insight into the structure of the film.

CPP 7.27 Mon 16:00 Poster B

Beitrag abgesagt — •XXX XXX —

CPP 7.28 Mon 16:00 Poster B

Nanostructure evolution during melting and oriented crystallization of polypropylene studied by in-situ SAXS — •Ulrich Nöchel[1], Armando Almendárez Camarillo[1], Norbert Stribeck[1], and Stephan Volkher Roth[2] — [1]University of Hamburg, Institute TMC, Hamburg, Germany — [2]HASYLAB at DESY, Hamburg, Germany

Polypropylene film with high uniaxial orientation and lamellar structure is melt-annealed at temperatures between 168°C and 175°C followed by different cooling programs during which 2D SAXS patterns are continuously taken in order to identify the mechanisms of crystallization. Peculiar "cross-patterns" observed during the early stages of crystallisation clearly demonstrate the mechanism of "building of lamellae from blocks" as proposed by Strobl. The evolution of the patterns under quiescent conditions shows that the complementary model of a "cross-hatched structure" cannot be applied. 2D SAXS data from various crystallization series[1] are evaluated by means of the CDF method[2] for a quantitative description of the crystallization mechanisms.

[1] Fibr. Text. EE (2005), 13(5), 27-29
[2] J. Appl. Cryst. (2001), 34(4), 496-503

CPP 7.29 Mon 16:00 Poster B

Optical diffraction measures the filling of superhydrophobic surfaces. — •Helmut Rathgen and Frieder Mugele — University of Twente, Physics of Complex Fluids, Postbus 217, 7500AE Enschede, The Netherlands

A hydrophobic surface with a periodic texture of period of order 1μm is a superhydrophobic surface and an optical grating at the same time. A drop deposited on such a superhydrophobic optical grating can float on the texture like a fakir (*Cassie-Baxter* state) or it can penetrate the texture, soaking the grooves of the grating completely (*Wenzel* state). In the prior case, micrometer size liquid-gas interfaces are formed at the rims of the grooves. The diffractive properties of the optical grating (diffraction efficiencies of all diffraction orders) now depend on position and curvature of these micro menisci. Realspace information can be obtained by comparing measured diffraction data to a theoretical model based on Multilayer Rigorous Coupled Wave Analysis. We use this to investigate filling state and microscopic curvature at a superhydrophobic surface for a true water-vapor two phase system as a function of external pressure. We find: 1. The surfaces fill (transition from Cassie-Baxter to Wenzel state) at a critical pressure that depends on geometry and hydrophobicity in agreement with a prediction based on capillary theory. 2. Filling cannot be reversed by decreasing the external pressure to the vapor pressure. Our method is relevant as a technique of optical detection for a range of problems in micro- and nanofluidics, such as tracer free liquid detection in nanochannels or surface cavitiation.

CPP 7.30 Mon 16:00 Poster B

Microemulsion-Polymer Systems Studied with Elastic and Inelastic Neutron Scattering — •Tinka Spehr[1,2], Bernhard Frick[1], and Bernd Stuehn[2] — [1]Institut Laue-Langevin, Grenoble, France — [2]Institute of Solid State Physics, TU Darmstadt, Germany

We study a microemulsion consisting of water, decane or toluene and AOT forming water-in-oil droplets. The addition of amphiphilic triblock copolymer leads to the interconnection of the droplets. We are investigating the structural and dynamical behavior of this model system for a transiently linked network with different neutron scattering techniques. Also this system is used to realize soft confinement of the water. Neutron Small Angle Scattering showed increasing ordering of the droplets upon polymer addition. Measurements on pure microemulsions have been carried out on a time-of-flight and a neutron backscattering instrument to study the effect of spatial soft confinement on water. Two different droplet sizes have been investigated (diameters of about 3 and 10 nm). Elastic scans from 315 K to 2 K showed a deeper undercooling of the water confined in the smaller droplets. Inelastic scans have been carried out at temperatures between 300 K and 250 K. The dynamic structure factor displays a complex shape clearly deviating from one single Lorentzian. The combination of spatial restriction and wall effects on the dynamics will be discussed. Neutron Spin Echo can probe shape fluctuations of the droplet shell [3]. We investigated the effect of polymer addition on the elastic properties of the surfactant shells.

[2] J. Huang, S. Milner, B. Farago, D. Richter (1987), PRL 59, 2600

CPP 7.31 Mon 16:00 Poster B

Water uptake and exchange kinetics of polyelectrolyte films: A neutron reflectometry study — •Regine v. Klitzing[1], John Wong[1], and Roland Steitz[2] — [1]TU Berlin, Stranski-Laboratorium, Straße des 17. Juni 124, D-10623 Berlin — [2]Hahn-Meitner-Institut, SF1, Glienicker Str. 100, D-14109 Berlin

The sequential layer–by–layer adsorption of polyanions and polycations to build polyelectrolyte multilayers has triggered enormous interest in their potential uses in a wide range of fields, from photonic to pharmaceutical applications. We will show that the conformation of the solvent swollen films – prior to drying – is determined by the initial adsorption conditions, but can be altered ex-situ by exposure to a liquid phase of very high ionic strength. Recently it has been observed that the swelling depends on the charge of the outermost layer. In the PAH/PSS system we saw that assemblies with PSS as the outermost layer swell more than those with PAH outside. A neutron reflectivity study of this effect in addition indicated the existence of two kinds of water, bound with different strength within the films. Beside an unexpected two-step kinetics of swelling, the reflectivity curves of the layers against vacuum before and after re–hydration in D2O vapor did not agree. It was only after subsequent re–hydration in saturated H2O vapor that the initial and final reflectivity curves against vacuum superimposed. We will discuss our findings in the context of polyion complex formation, interdigitation and film imperfections.

CPP 7.32 Mon 16:00 Poster B

Horizontal ToF-Neutron Reflectometer REFSANS at FRM II Munich: Potential and First Experimental Results — •Reinhard Kampmann[1], Martin Haese-Seiller[1], Valeri Kudryashov[1], Bert Nickel[2], Peter Müller-Buschbaum[4], Christine Papadakis[4], Wilhelm Fenzl[2], Andreas Schreyer[1], Erich Sackmann[3], and Joachim Rädler[2] — [1]GKSS-Forschungszentrum Geesthacht GmbH, D-21502 Geesthacht, Germany — [2]Department für Physik, Ludwig-Maximilians-Universität, D-80539 München, Germany — [3]Physik-Department E22, TU-München, D-85748 Garching, Germany — [4]Physik-Department E13, TU-München, D-85748 Garching, Germany

The reflectometer REFSANS allows to perform comprehensive analyses of vertical and lateral surface and interface structures by means of specular and off-specular neutron reflectivity as well as small-angle neutron scattering at grazing incidence (GISANS). All measurements can be performed on the air-water interface (horizontally aligned sample). REFSANS has been put into operation in 2005/06. Its performance is demonstrated by results of first reflectivity as well as GISANS measurements on nanostructured polymer and metallic films. The potential of this novel instrument for measuring weak off-specular scattering, GI-SANS and extremely low specular reflectivity including the case of a strongly incoherently scattering substrate is discussed.

This work has been supported by the German Federal Ministry of Education, Research and Technology (BMBF) under contracts 03-KA5FRM-1 and 03-KAE8X-3.