

Abstract book

2nd Workshop "GISAXS - An Advanced Scattering Method"

organized by

P. Müller-Buschbaum (TU München) S.V. Roth (DESY) R. Gehrke (DESY)

May 09, 2007 - May 11, 2007 at HASYLAB / DESY, Notkestr. 85, D-22607 Hamburg (Germany)

Buildg. 28c

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2nd Workshop "GISAXS - An Advanced Scattering Method"

May 09, 2007 - May 11, 2007 at HASYLAB / DESY, Notkestr. 85, D-22607 Hamburg (Germany)

Programme

Wednesday, May 09, 2007

08:00-09:00 Registration

Section 1 – Lectures

Welcome

09:00-09:30 Current and Future Photon Science at DESY *R. Gehrke (DESY Hamburg, Germany)*

Session Modelling

09:30-10:15	Theoretical Foundations of GISAXS
	M. Rauscher (MPI Metallforschung, Stuttgart, Germany)
10:15-11:00	Simulation of Grazing Incidence Small-Angle X-ray
	Scattering from Nanostructures
	R. Lazzari (Univ. Paris, France)

Break

Session Quantum Objects

11:30-12:15	GIXD on Quantum Dots
	T. Metzger (ESRF Grenoble, France)
12:15-12:45	GISAXS Studies on Dielectric Thin Films
	J.P. Simon (CNRS & Univ. J. Fourier Grenoble, France)
12:45-13:15	GISAXS on Quantum Dots
	H. Okuda (Univ. Kyoto, Japan)

13:15-14:45 Lunch

Session Polymer-Engineered Thin Films

14:45-15:30 Kinetic Investigations in Thin Polymer Films

	A. Gibaud (Univ. Le Mans, France)
15:30-16:00	Pushing the Limits of GISAXS – Nanobeams
	and µm-Length Scales
	P. Müller-Buschbaum (TU Munich, Germany)
16:00-16:30	GISAXS on Ordered Polymer Films
	G. Fritz (Univ. Graz, Austria)

Break

17:00-19:00	Poster Session (contributed presentations)
19:30-22:30	Workshop Dinner

Thursday, May 10, 2007

Session Biophysics & Sensors

09:00-09:45	Interaction between peptide pores in lipid bilayers
	D. Constantin (Univ. Paris-Sud, Orsay, France)
09:45-10:15	Surface Sensors Investigated with µGISAXS
	J.S. Gutmann (MPI f. Polymer research, Mainz, Germany)

Break

Session Nanocomposite thin films

10:45-11:30	Nanostructured Thin Films
	M. Ree (Univ. Pohang, Rep. of Corea)
11:30-12:00	GISAXS on Advanced Nanostructured Materials
	A. Frömsdorf (Univ. Hamburg, Germany)

Session Instrumentation

12:00-12:30 12:30-13:00	Introduction to BW4 A. Timmann (DESY Hamburg, Germany) GISAXS at BW4 & Future Possibilities S.V. Roth (DESY Hamburg, Germany)
13:00-14:30	Lunch
14:30-15:00	30 min Safety Course S.V. Roth (DESY Hamburg, Germany)
15:00-15:15	Organization into Groups

Section 2 - Practical training

3 groups up to 10 persons, rotating between (1), (2), (3) each part: 3h with including 20min break to change between stations

15:30-18:30	Practical training part 1
	(1) Measurement @ BW4

	Tutors: (2) Data treatment: (3) Simulation:	P. Müller-Buschbaum / S.V. Roth J.S. Gutmann / A. Timmann R. Lazzari / R. Gehrke
18:45-20:45	Poster Session (cor Refreshments	ntributed presentations)
	Friday , May 1	1, 2007
09:00-12:00	Practical training pa	urt 2
12:00-13:00	Lunch	
13:00-16:00	Practical training pa	irt 3

Conference website: https://indico.desy.de/conferenceDisplay.py?confld=111

Abstracts of invited speakers

Interaction between peptide pores in lipid bilayers

D. Constantin, G. Brotons¹, A. Jarre², C. Li² and T. Salditt²

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The elucidation of lipid-mediated interaction forces between membrane proteins and the corresponding lateral distribution in the plane of the membrane is an important step towards a quantitative understanding of the functional mechanisms of membrane proteins and membrane peptides. Experimentally, the lateral structure and organization of multi-component membranes is as important as it is difficult to probe. In this work we show how the lateral and vertical intensity profiles of a peptide pore correlation peak can be analyzed as a function of peptide concentration to determine the corresponding interaction forces.

A well-known example of biological function deriving from lipid-peptide interaction and selfassembly is the activity of a family of short and amphiphilic membrane active polypeptides denoted as antimicrobial peptides. These molecules bind to microbial cell membranes, subsequently causing an increase in membrane permeability and cell lysis. One such molecule is alamethicin, a 20 amino acid peptide from the fungus *Trichoderma viride*; it is well known that alamethicin acts by creating pores in the cell membrane. Pore formation is usually a highly cooperative process; this was confirmed for alamethicin and a membrane-mediated interaction between peptides was invoked to explain the phenomenon.



Figure 1: Structure of the alamethicin pore (from [1]). Top view (left) and side view (center). Electron density profile (right) reconstructed from the atomic coordinates of an MD simulation [2].

Although determining the interaction between (adsorbed or inserted) monomers is very difficult and, to our knowledge, has never been attempted, the interaction between already formed *pores* within the membrane can be studied using neutron or X-ray scattering from oriented multilamellar stacks, a method pioneered by Huang and collaborators [3-5]. For the case of alamethicin, they observed a lateral correlation peak, which was attributed to liquid-like ordering of pores in the plane of the membrane and was modelled based on hard disk interaction, with very satisfactory results. However, in these studies at most two peptide-to-lipid concentrations P/L were investigated for each system.

Building upon this work, we gathered detailed information on the quasi two-dimensional fluid of pores in the lipid bilayer, using high-resolution synchrotron scattering from aligned multilamellar stacks of alamethicin/DMPC mixtures. We measured the two-dimensional scattering distribution for an entire concentration series P/L and performed a simultaneous lineshape analysis on all recorded curves.

We found that the in-plane interaction potential consists of a hard core, with a radius that agrees very well with the geometrical outer radius of the pore, and an additional repulsive contribution which can be described as a Gaussian, with a range of 31.5 Å and a contact value of 2.41 k_BT . The results are in qualitative agreement with recent theoretical models [6-7].



Figure 2: a) Scattering from the 2D fluid of alamethicin pores as a function of the wave vector for different peptide-to-lipid concentrations (symbols) and fit with the theoretical model (lines). b) The interaction potential used for the fits in a). It consists of a hard core with a diameter 2R = 36.4 Å and an additional Gaussian repulsion. c) Pictorial representation of the fluid of pores in the plane of the bilayer: alamethicin monomers (red), inner water pore (blue) and repulsive potential (gray halo).

These results are accepted for publication in the *Biophysical Journal* [8].

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GISAXS on Ordered Polymer Films

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Ring opening metathesis polymerization (ROMP) is a powerful polymerization method of synthesizing block copolymers with low polydispersity indices. We used polymers based on blocks of *endo,exo*[2.2.1]bicyclo-2-ene-5,6-dicarboxylic acid dimethylester and *endo,exo*[2.2.1]bicyclo-2-ene-5,6-dicarboxylic acid. Such block copolymers form structures in bulk but also within thin films on substrates. The structures within these films were studied using GISAXS, while molecular weight, relative block-length, type of film preparation technique, solvent, and substrate were varied. The film structures are very similar for most substrates, although Teflon induces a considerably different order. A much stronger influence is caused by the structure of the polymers in solution, i.e., if the solvent is specific or non-specific for the two blocks. The results are compared to the solution structures studied by SAXS and DLS and to the bulk structures investigated by SAXS and TEM..

GISAXS on Advanced Nanostructured Materials

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Nano-scale structures offer a great potential for advancements in electronics, opto-electronics, high-density storage media, catalysis and biological applications. These nano-sized materials display properties that differ from their respective bulk material counterparts. In this talk the preparation and charaterization of different nanostrucured materials are presented. After an introduction, how the DWBA influences the GISAXS intensities, a comparative experiment and simulation of two types of two-dimensional arrays of PMMA beads and their influence on the scattering patterns is shown [1]. In another experiment highly ordered 3-D supernanocrystals of PbS [2] and also highly ordered 2-D arrays of CoPt₃ were produced. The temperature degradation of CoPt₃ was followed in a GISAXS experiment and analysis and simulation will be discussed. Furthermore, the size-controlled preparation of silica and vanadium oxide filled micelle cores and their self assembly behavior are presented [3]. In a second step it is possible to use these structures as templates for highly ordered magnetic nanostructures, revealed by Ar+ ion milling [4]. The resulting structures were characterized by different imaging and scattering techniques and model simulations were performed. First results from in-situ GISAXS experiments while DeskJet-printing nanostructures are shown. With this method one is able to produce superhydrophobic surfaces or nanoparticle arrays with a specified height. The characterization of the obtained nanostructured surfaces has be done by atomic force microscopy (AFM), by scanning electron microscopy (SEM), grazing incidence X-ray small angle scattering (GISAXS) and X-ray reflectivity measurements (XRR). For the investigation of the magnetic properties the magneto optical Kerr effect (MOKE) was utilized



Figure 1: Variety of nanostrucured surfaces obtained by block copolymer lithography and/or nanoparticle depositon using different techniques

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Current and Future Photon Science at DESY

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Simulations of Grazing Incidence Small Angle X-rays Scattering from nanostructures

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The aim of this talk is to give an overview of the capabilities of the *IsGISAXS* software [1] to deal with simulations and fits of GISAXS from nanostructures [1]. It will be shown that the treated geometries of layers of nanoparticles (islands on a surface, holes in a substrate, inclusions in a layer etc...) allow separating in the scattering cross section the in-plane interferences from the particle form factor. This latter includes multiple scattering effects due to the grazing scattering geometry. For disordered collections of particles, the emphasis will be put on the complex interplay between coherent and incoherent scattering through a review of the various used approximations. The influence of the exact profile of refraction index and of the correlation between particle sizes and spacing on the scattering will be discussed. Two examples of data analysis will illustrate the talk:

- (i) the self similar growth of Au nanoparticles on $TiO_2(110)$;
- (ii) the self organized growth of Co particles on surfaces patterned by a buried interfacial dislocation network.



Figure 1: Experimental and simulated GISAXS pattern from Au nanoparticles ($R\sim1.5nm$) grown *in situ* by vapour deposition on TiO₂(110).

References

[1] IsGISAXS web site : http://www.insp.jussieu.fr/axe2/Oxydes/IsGISAXS/isgisaxs.htm

GISAXS and GID:

What is there to learn about Semiconductor Nanostructures

T. H. METZGER

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Following Moore's law the continuous shrinking of device sizes is approaching the nanoscale. The nanoscale is not just another step towards miniaturization. It is a qualitatively new scale where materials properties, such as melting point, electrical conductivity, optical properties differ significantly from the same properties in the bulk.

For quantum dots, these properties can be tuned by structural parameters like size, shape, strain, composition and atomic ordering. In order to understand the growth and functionality of quantum dots, these parameters must be characterized on an atomic level.

X-ray scattering techniques, based on synchrotron radiation, are well suited to investigate these nanostructures. In this talk it will be demonstrated that the combination of grazing incidence small angle scattering (GISAXS) and diffraction (GID) allows for a determination of size, shape and internal strain in the quantum dots. Combined with the element specific anomalous dispersion of the atomic scattering factor, these methods are transformed into chemical sensitive tools. Thus, 3D chemical composition in quantum dots can readily be derived.

In this lecture I will demonstrate the complementary of GISAXS and GID to be ideally suited to combine the information on morphology and ordering of nanostructures with the crystalline properties such as strain, chemical composition and atomic ordering (in case the nanostructures are composed of an alloy). I will first briefly describe the beamline ID01, which is specialised to combine GISAXS and WAXS (GID) simultaneously and change the x-ray energy at fixed exit, in order to perform these techniques in an "anomalous way".



Figure 1: in-situ GISAXS after growth of more than 6 ML of Ge on Si(001) (courtesy of M.I. Richard, T. Schuelli, G. Renaud)

I will then present growth results on the Straski-Krastanov system Ge on Si measured by in-situ GISAXS, where the evolution of the islands and facet formation can be followed as a function of the number of Ge layers deposited (figure 1).

In the same system we have measured the 3D form factor of truncated pyramids (grown by LPE) where the 3D intensity distribution reveals strong streaks perpendicular to the 111 facets and 2D sheets perpendicular to the edges of the pyramids. Using DWBA all features can be explained and quantified.





In order to study the crystalline properties of nanostructures, i. e. strain and composition, we have developed the Iso Strain Scattering (ISS) technique. The first "historical measurements" were done at Hasylab, beamline BW2 (Figure 2). ISS has been combined with anomalous scattering and has been employed to study many different quantum dot systems with great success.

The ISS will be explained and latest results on Ge islands on Si(001) will be presented. Anomalous ISS experiments have shown that a considerable amount of Si from the substrate is incorporated into Ge dome-shaped islands during growth [2]. In order to investigate the degree of atomic ordering in this alloy, basis forbidden reflections were measured in nominally pure Ge islands on Si(001) [3].

The last example is on ion beam sputtered (IBS) semiconductor surfaces, which is an alternative method for the controlled production of nanostructures on a surface. The formation and evolution of the nanostructures formed on GaSb(100) has been studied in-situ by means of grazing incidence X-ray scattering techniques.

The outlook of my talk is dedicated to the ongoing refurbishment of ID01aimed to obtain a stabile coherent beam suitable for coherent diffraction imaging and submicron diffraction on single nanostructures.

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Theoretical Foundations of GISAXS

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Small angle X-ray scattering under gracing incidence is a powerful tool that allows for high resolution surface or interface sensitive structural analysis. While the theoretical basis of most scattering techniques is the Born Approximation (BA, well discussed in quantum mechanics text books such as, e.g., [1]) based on plane waves as basic solutions of the wave equation, under gracing incidence, multiple scattering becomes important. This, for example, leads to total reflection of photons if the angle of incidence is small enough; a phenomenon well known for visible light.

Fortunately, a full dynamic scattering theory is not necessary. The most important dynamical effects can be taken into account in a improved version of the BA, the Distorted Wave Born Approximation (DWBA, see for example [2]). The idea is to "split" the sample from which the X-rays are scattered into a simple system for which the wave equation can be solved analytically (e.g., for a sample with islands on top this would be a flat and planar interface as illustrated in Figure 1, see [3]) and a perturbation (the islands), the scattering from which can then be treated kinematically (i.e., neglecting multiple scattering events).



Figure 1: The basic idea of DWBA. A complicated sample (e.g., a substrate with islands on top, left side) is "split" into a simple system (the flat substrate, bottom right) and a perturbation (the islands, top right).

In the talk I will the mathematical and practical aspects of DWBA starting with a short review of the BA. For the paradigmatic example of a rough surface I will develop the DWBA in detail, covering the origin and nature of the Yoneda peak, the role of coherence, and the limits of the method [4]. In the second part of the lecture, I will briefly show how the DWBA can be applied to more complicated systems such as rough multilayers [5], islands on a surface[3], and thin films with an internal structure [6].

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Analysis of mesostructured thin films by X-ray reflectivity and GISAXS

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After an introduction of the two techniques, I will present some applications of such techniques to the study of gratings and mesoporous materials. For gratings, I will present how the intersection of grating truncation rods (GTR) with the Ewald sphere can be determined. As shown in Figure 1, the influence of the misorientation of the grating about the azimuthal angle will be shown and the location of the diffracted spots will be analyzed. In addition the anisotropy of the width of these spots will be discussed. [1,2]



Figure 1: Illustration of the grating truncation rods with the Ewald sphere when the grating is rotated by 1° about the azimuthal angle (left panel, $\phi=1^{\circ}$) and when it is almost aligned with the direct beam (right panel,

 $\phi=0^{\circ}$). The crosses are obtained by simulation. (period of the grating 440nm).

I will then move to the study of mesoporous materials synthesized by supramolecular self-assembly of surfactant molecules to template the condensation of inorganic species. The use of GISAXS and X-ray reflectivity will be presented as unique tools to investigate in a non

intrusive way the structure a d the electron density profile of such materials. The GISAXS technique is used to ascertain the structure of the films (see Figure 2) and its evolution upon the removal of the surfactant.



Figure 2 : Typical GISAXS patterns of a 2D hexagonal structure of a silica thin film templated by the pluronics P123 triblock copolymer (left panel before removal of the surfactant, right panel after the removal).

Note the change of intensity of Bragg reflections upon removal of the surfactant.

This will give me the opportunity to show how one can derive the porosity of such

films. In addition in-situ experiments of capillary condensation inside the pores will be described and I will show how the Young modulus of such materials can be apprehended by

the x-ray reflectivity technique. [3,4,5].

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Microstructures of self-assembled nanodots

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Local stability of inorganic nanostructures at or near the sample surfaces, such as semiconducting nanodots capped with similar or dissimilar materials, or metallic nanodots grown on flat surfaces has drawn attention these decades. Many preceding works have been published on the microstructures of self-assembled semiconducting[1]-[4] and metallic nanostructures[5]. Concerning semiconducting nanostructures, morphology and stability of such nanodots are particularly interesting from a viewpoint of thermal stability and control of heterointerfaces. For example, the intermixing at the heterointerface was a serious problem already for the multiple quantum well (MQW) structures, and has been examined by, for example, CTR scattering and direct cross-sectional STM[6]. Although the stability of interfaces is also important for quantum dots, it is rather difficult to evaluate. We have examined the microstructure of Ge nanodots encapsulated by a Si layer and InAs compound semiconductor nanodots encapsulated by an As layer by GISAXS. The effect of annealing on the capped semiconducting nanodots are examined by SAXS and reflectivity measurements. For Ge nanodots, isothermal post annealing at a temperature about 40 K higher than the growth temperature showed a small blue shift of photo luminescence peak wavelength, corresponding to an increase of interdiffusion layer at the interface. On the other hand, the shift in the PL peak is also observed for some isochronally annealed samples, where the interdiffusion destroyed the quantum dot structures and the blue shift simply reflects a change in the average composition by interdiffusion. Although these problems are discussed by a combination of GISAXS and diffraction[4,8], quantitative assessment is still challenging. For compound semiconductor nanodots, growth of GaAs cap layer induces much interdiffusion than that of Si on Ge nanodots.



Figure 1: GISAXS patterns of InAs nanodots encapsulated with amorphous As layer. The dots began to have facets of almost {113} planes after the average InAs layer exceeds 2 monolayers.

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Investigation of micro mechanical cantilever sensors with Micro-focus SAXS

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Micro mechanical cantilevers covered with polymer brushes are of great interest for fundamental research and practical applications [1]. Used in fundamental research they allow an insight into the swelling behaviour of polymer brushes. Furthermore they can be used for a wide variety of sensor applications. In priciple a micro mechanical cantilever sensor consist of two main parts: the micromechanical cantilever that acts as a transducer element and a coating which can be highly specific for different molecules and produces specific response patterns for various analytes [2]. The polymer brushes used in our experiments have several advantages compared with physisorbed coatings: Polymer brushes are covalently bound onto the surface. Therefore this sensor type can be used within gases and liquids as well, without damaging the sensitive layer due to desorption. Another advantage is a higher sensitivity and a stronger response to an external stimulus due to the internal osmotic pressure of the polymer brushes.

As transducers OCTOSENSIS silicon microarrays (Micromotive, Germany) (see figure 1) are typically used. Each chip consists of eight rectangular cantilevers, with a length of 750 μ m, width of 90 μ m and thickness of 1 μ m arranged at a pitch of 250 μ m.



Figure 1: Micro mechanical cantilver array with 8 cantilevers.

In a typical sensor functionalization a protective gold layer with a thickness of 20 nm is deposited on part of the cantilever in order to preserve it as reference. The gold coating is

applied on the entire backside and on the half of the topside of the array. On the other half a starter was attached on the uncovered surface of the cantilver array. Utilizing an atom transfer polymerisation (ATRP) "grafting from" technique Poly(methyl methacrylate), PMMA, brushes is then grown on the silinated array surface [3]. An average number molecular mass, M_n of 51 kg mol⁻¹ was measured via GPC for the bulk PMMA that was synthesized in the same process.



Figure 2: Intensity of the specular peak scanned across the cantilver array. From the signal height we can conclude about different coatings: a) gold b) gold contamination and polymer brushes c) polymer brushes.

For sensing applications, the quality of the coating, grafting density of the polymer brushes, layer thickness, uniformity of the layer are very important for the response of the sensor on an external stimulus. Using microfocus experiments we investigated the quality of the cantilever coatings, especially the quality of the gold layer and proofing the existence sharp boundaries between the polymer coated cantlivers and the ones gold coated. The experiments were carried out at the BW4 beamline using a μ -Focus-GISAXS setup. The the detector sample distance was typically $l_{sd} \approx 1.9$ m and the dimension of the micro-focus beam varied between 35 x 60 μ m and 17 x 32 μ m. In figure 2 were the intensity of the specular peak scanned across the cantilver array is shown. We can conclude from the signal height about different coatings: In the yellow marked region we achieved a pure gold layer on top of the cantilevers, while in blue region only polymer brushes are present [4]. In addition we found an intermidiate region (marked orange) with polymer brushes contaminated with gold. Additionally the μ -GISAXS experiments allowed us to determine film thickness and lateral structure of the polymeric coatings via the selective recording of a scattering information from a single microcantilever.

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Pushing the Limits of GISAXS - Nanobeams and micro-Length Scales

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Over the past years, grazing incidence small angle x-ray scattering (GISAXS) turned out to be a powerful advanced scattering technique for the investigation of structured polymer films [1]. While bulk samples are routinely probed by small angle x-ray scattering (SAXS), the grazing incidence geometry enables the necessary surface sensitivity. In contrary to common real space analysis techniques such as scanning force microscopy or electron beam based microscopy GISAXS is a reciprocal space analysis technique which enables the detection of surface and buried structures in a non destructive way. In addition, GISAXS does not require a special sample preparation and thus enables kinetic investigations devoted to a structural development as a function of time. As compared to local probe techniques, GISAXS yields an excellent sampling statistics, which means that it averages over macroscopic regions to provide information down to a nanometer scale. Based on a detailed modeling of the data, the statistical information results in the detection of an object geometry, size distributions and spatial correlations between objects, very comparable to what is possible in SAXS. [2]

Recent experimental improvements, which overcome both restrictions of GISAXS, the necessary sample homogeneity and the quite small upper limit of the detectable structural size, are focussed. Both presented experimental developments of GISAXS rely on focussing the X-ray beam. The focussing of the beam onto the sample position results in a high real space resolution at the position of the sample. As a consequence, micro-focussed beams combined with GISAXS and scanning of the sample with respect to the beam allow to probe local structures instead of homogeneous samples [3-5]. In contrast, focussing the X-ray beam on the detector yields a high reciprocal space resolution, which is necessary to probe large scale structures [6-8]. Such high resolution experiments in the GISAXS geometry are frequently named with GIUSAXS in analogy to the transmission geometry.



Figure 1: a) Example of blend film sample measured under two different experimental resolution conditions: high resolution set-up (blue open circles) and ultra-high resolution (red crosses). The corresponding resolution limits are marked by vertical lines and the most prominent in-plane length of 4.8 µm is marked by an arrow 'A'. The solid lines are fits based on a model described in the text. b) Corresponding optical micrograph.

The possibilities of both types of advanced GISAXS set-ups are demonstrated by staying in the model system of a binary polymer blend film. Blending different polymers and thereby obtaining new and tailored material properties is one of the bases of the success of polymer applications. One prominent example is the tailoring of mechanical properties by mixing soft and hard polymer components, or combining a polymer with a glass transition temperature one well above room

temperature with one well below. Coating solid surfaces with such polymer blends and conserving the properties of the bulk blend material in the blend film is an extremely attractive way to obtain functional coatings. Large scale phase separation structures are typically formed due to the immiscibility of the blended polymers, ensuring a variety of applications, e.g. in optics or sensors. The morphology is altered and tailored by changing the ratio of the components in the blend. The characteristic structural size is modified through the amount of material deposited on the solid support and by the preparation technique applied. Large scale structures are an intrinsic feature of such blend films (see figure 1). The sample heterogeneity is introduced by the superposition of phase separation and flow.

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Quantitative GIXS Analysis of Nanostructured Thin Films

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A schematic optical setup of grazing incidence X-ray scattering (GIXS) is given and compared with that of conventional transmission X-ray scattering (TXS) in Fig. 1. Synchrotron GIXS has several important advantages over transmission X-ray and neutron scattering as well as scanning and transmission electron microscopies: (i) a highly intense scattering pattern is always obtained, even for films of nanoscale thickness as well as nanostructures on substrates, because the X-ray beam path length through the film plane is sufficiently long; (ii) there is no unfavorable scattering from the substrate on which the film is coated; and (iii) easy sample preparation [1-11]. For these advantages, in recent years the GIXS has become the major analytical tool for characterizing structures and properties of a variety of nanostructures and nanoscale thin films in a single and multilevels. However, the quantitative analysis of measured GIXS data requires developing a new proper scattering theory because of the complexity of GIXS phenomenon due to the scatterings from the transmission and reflected X-ray and the refraction effect, which is very far from the conventional transmission and reflection X-ray and neutron scattering. We have applied this powerful GIXS technique to quantitatively characterize nanostructured thin films. In our study GIXS measurements with synchrotron radiation sources were conducted statically and in-situ for a series of nanoscale thin films prepared from nanoporous dielectrics, block copolymers, brush polymers, and molecular assemblies (Figs. 1, 2, and 3) [1-11]. All GIXS measurements were performed at the Pohang Accelerator Laboratory (PAL) (Fig. 4) [12]. The measured scattering data were analyzed in detail by using the newly developed GIXS scattering theory. All GIXS results will be discussed in details with considering the materials chemistry and nanostructure formation process parameters.

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Fig. 1. Schematic diagrams of GIXS and TXS.Fig. 2. AFM and GIXS results of
PS-b-PI film.Fig. 3. Thermal phase transition of a
PS-b-PI film.



Fig. 4. Synchrotron facility of Pohang Accelerator Laboraory (Pal) at Pohang University of Science & Technology (Postech)

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GISAXS at BW4 & Future Possibilities

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In recent years, grazing incidence small-angle x-ray scattering (GISAXS) has been strongly developing as an advanced method for surface and interface characterization, including in-situ studies. The wiggler beamline BW4 / HASYLAB has therefore established a dedicated GISAXS mode offering several opportunities. This includes high reciprocal space resolution GIUSAXS [1] as well as microbeam GISAXS using a microfocused x-ray beam [2]. μ GISAXS and GIUSAXS are routine methods at BW4.

Among the first beamlines to be built at PETRA III is the microfocus small- and wide-angle x-ray scattering beamline μ SAXS/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 μ GISAXS [2,3] and microbeam SAXS tomography [4]. 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-40 μ m, thus allowing to adapt the beam size to the demands of the scanning experiments. The design of the beamline especially allows for combining ultra small angle-xray scattering (USAXS) with a microfocused beam (μ USAXS). I will present the present layout of the μ SAXS/WAXS beamline introducing the projected capabilities of this new micro- and nanofocus scattering beamline at PETRA III.

As an application example, I will focus on Polymer-based nanocomposites. Such nanocomposite materials allow to combine the extraordinary features of the individual materials forming the nanocomposite. Especially the richness of polymer structures in blend as well as blockcopolymer systems in thin film geometry gives rise to new classes of nanocomposite materials. Annealing as a second step in nanocomposite preparation allows for further nanostructuring the polymer film inducing e.g. a definite roughness or particle distribution. In our approach we combine the richness of the polymer structure with a metal coating [3,5]. Such polymer-based metal nanocomposites are widely used in many optical and biotechnological applications. As experimental method to study such nanocomposites, we used microbeam grazing incidence small-angle x-ray scattering at the beamline BW4 of HASYLAB (Hamburg). Our results reveal the dominating influence of the polymer layer in the structure of this nanocomposite.

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GISAXS studies of dielectrics thin films for ultra large scaled integrated microelectronics

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The increase of the integration density and of the operation speed in ultra large scaled integrated microelectronics requires to reduce the dielectric constant for high frequency insulation between the copper connections of some tenth-of-micrometer thickness. The quality of the dielectric is defined by its dielectric constant k (> 1) relative to the unpolarized vacuum (k = 1). Bulk low k will never reach k lowed than 3 and the only way to achieve further k decrease is to introduce nanopores dielectric films compatible with required mechanical behaviour [1]. Ree & Lee [2, this workshop] will show how Korea has tackled this problem.

For deposited layers much thicker (~300nm) than the size of the scattering objects (<10nm), GISAXS analysis of the corrected data is the same as the one of SAXS in transmission. GISAXS allows to measure layers deposited on opaque buffers and, due to the grazing angle (above the critical angle, in order that the beam penetrates into the layer), increases the signal in $1/\sin(\alpha_i)$, α_i being the grazing angle, i.e. of ~300. The drawbacks are: i) the measure needs a synchrotron beam (in the present case the anomalous scattering/diffraction beamline, D2AM, at ESRF, ii) due to the shadow oof the sample for α_i in the range 0.1-0.5 degree at 8keV, we are unable to measure scattering object sizes large then ~10nm in the direction perpendicular to the layer. In a second step, corrections of reflexion, refraction and transmission are needed [3]. Finally comparative (or absolute) intensities can only be extracted if the beam monitoring do correspond to the beam impinging the sample: thanks to our 70mm long samples and our 0.1mm beam height, all the beam is intercepted by the sample and a correction of the intensities in $1/\sin(\alpha_i)$ give a superimposition of the SAXS patterns for different α_i [4]. We compare the merits and the structure determined of different growth processes, porogen approach, self assembled and PECVD. All of them are baked in order to cure the amorphous Si_wO_xC_yH_z "skeleton" (SiOCH). Depending on the process used, the pore morphologies are very different [4]: they range from well-defined pores of 4-5 nm of diameter, with occasionally an anisotropy of the pattern, to sub nanometric ill-defined pores which may be described as density fluctuations. The sizes and volume fraction f_v can be compared to two other techniques: elipsometry-porosimetry [5] and X-ray Reflectivity. Finally, it appears that the curing process is a key problem, which up to now has been difficult to characterize by GISAXS.

Acknowledgements: The presented exemples are taken from the long term collaboration with V. Jousseaume and G. Rolland [1, 4, 5]. Thanks also to the D2AM team Drs Bérar, Boudet and Caillot.

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Scattering experiments at the BW4

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The BW4 is a dedicated SAXS beamline, which has the capabilities for normal SAXS as well as für advanced techniques like USAXS and GISAXS. The real space resolutions are 2 nm to 200 nm in SAXS and 1 nm to 400 nm in GISAXS. With sample to detector distances of up to 13.5 m the upper limits for USAXS and GIUSAXS are 1 μ m and 13 μ m, respectively. There is also a moderate micro-focus option available which is produced by a Beryllium-compound refractive lens (Be-CRL). With this a beam size down to $32 \times 17 \,\mu$ m² (H×V) possible. Special sample environments provided are heating stages for transmission and grazing incident measurements as well as a stretching device. [1]

To show the feasibility of such optics there was a temporary setup was a USAXS measurement with a Be-CRL as focusing optic. This was done without mirrors and gave a total flux of 5×10^{11} photons/s at sample position. Figure 1 shows the scattering pattern of a collagen fibre with this setup at a sample detector distance of 12.6 m.



Figure 1: Scattering pattern(left) and averaged scattering curve(right) of a collagen fibre measured with USAXS using a BeCRL as focusing optic.

Another experiment conducted at the BW4 was an in-situ GISAXS measurement of a colloid solution unter shear stress. A 13 wt.% solution of a poly-(isoprene-block-ethylene oxide) in water forming a FCC lattice was investigated. The block degrees of polymerization of the polyisoprene and the polyethylene oxide were 55 and 170, respectively. [2] The shear stress was applied by a stress-controlled Bohlin CVO rheometer in a plate-plate-geometry with a diameter of 20 mm and a gap of 1 mm. We investigated the interface layer of the solution with the rotor of the shear geometry. The measurements were conducted at different temperatures. The figure 2 shows the result of the measurements at a temperature of $T = 15^{\circ}$. This is representative for all temoperatures. We found that the layer thickness of the metal-solution interface increases with the first application

of shear. After that the increase of the shear rate doesn't change the thickness of the layer anymore. From this we conclude that the micelles deposited at the metal surface get ripped of by the application of shear stress and then stay at a constant distance to the surface.



Figure 2: Scattering patterns at the rotor wit increasing shear (a). The white lines indicate the detector cuts without shear stress and at $\tau = 120 \text{ Pa}$ (b).

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Poster presentations

Alphabetical List of Poster presentations

A1. G.A. Abbas, P. Papakonstantinou, and J. McLaughlin University of Ulster, NIBEC, Shore road, Co. Antrim, Northern Ireland, Newtownabbey GB

"Synchrotron based X-ray characterisation of stress-free amorphous carbon thin films"

A2. M. M. Abul Kashem, J. Perlich, L. Schulz, S. V. Roth* and P. Müller-Buschbaum TU München, Physik-Department LS E13, James-Franck-Str. 1, 85747 Garching, (Germany) * HASYLAB at DESY, Notkestr. 85, 22603 Hamburg (Germany)

"Investigation of structures in diblock copolymer film containing magnetic nanoparticles"

Nanocomposite materials based on polymeric matrix and inorganic fillers having desired mechanical, electrical and thermal properties have been being reported since long. Rendering magnetic properties to the polymer by using magnetic nanoparticles is a rather new idea. We have investigated the nanostructures supported by superstructures formed in ultra thin films of such a new type of nanocomposite materials. The system is a diblock copolymer p(styrene-block-isoprene) matrix with embedded magnetic nanoparticles (metal oxide) covered with polymeric (PS) hairs. The superstructure is formed due to dewetting of the polymer film and the nanostructure due to self-assembly of diblock copolymer. We were able to produce dewetted structures containing magnetic nanoparticles. The nanoparticles retard the dewetting process and control the orientation from a perpendicular to a parallel lamella. Moreover, roughness correlation between the substrate and the film surface has been observed at different film thicknesses resulting from gradient sample. The investigation has been carried out by atomic force microscopy (AFM) in non-contact mode and by using micro-beam grazing incident small angle X-ray scattering (microGISAXS).

A3. Byungcheol Ahn, Kyuyoung Heo, Weontae Oh, Jinhwan Yoon, Byeongdu Lee, Yongtaek Hwang, Jong-Seong Kim, Young-Hee Park, and Moonhor Ree

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"Grazing Incidence X-ray Scattering Studies on Templating Nanopores in Networked Polymer Thin Films with a Multi-armed Porogen"

Porous organosilicate materials have recently attracted much interest due to their potential application as low dielectric constant (low-k) interdielectrics. In particular, much effort has been directed towards the development of low-k porous organosilicate dielectric thin films via the templated polycondensation of their soluble precursors in the presence of a thermally-labile, organic polymeric porogen. Pores are subsequently formed in the resulting dielectrics through the sacrificial thermal decomposition of the porogens in the range 350-400C. However, the tendency of porogens to aggregate in organosilicates has limited the ability to reduce the pore size and porosity of the resulting dielectrics, making them unsuitable for use in advanced integrated circuits patterned with small feature sizes. The formation of porous films from an organosilicate precursor polymer/porogen composite is a complex procedure since the matrix precursor readily undergoes crosslinking while the porogen undergoes thermal degradation. In order to understand the structure of pores in the porous film, it is necessary to investigate their generation during the porous dielectric film process. Pore structure evolution during porous film process has rarely been investigated. In this study, we report the in-situ grazing incidence X-ray scattering (GIXS) study of the evolution of nanopores during porous dielectric film formation in a composite film containing an organosilicate precursor and six-armed star shape poly(-caprolactone) porogen in various compositions. Using a synchrotron X-ray source, in-situ GIXS measurements were carried out during thermal treatment of the organosilicate/porogen composite films to 400 C in vacuum, and continued during the subsequent cooling of the resulting porous films. In addition, thermogravimetric analysis and transmission electron microscopy measurements were performed. Detailed analyses of the measured two-dimensional (2D) GIXS data was ac complished using a GIXS theory derived under the distorted wave Born approximation. The following structural characteristics of both the composite films and resulting porous films: miscibility of precursor matrix and porogen, phase separation, pore generation mechanism, pore size and size distribution, film electron density, and film porosity, were also determined. All results will be discussed in detail with considering chemistry, miscibility, curing reaction, hybridization, decomposition, phase changes, and process conditions.

A4. M. Al-Hussein Physics Department, University of Jordan, Amman, Jordan

" Investigating the nano structure of self-assembling amorphous/liquid crystalline diblock copolymers using grazing incidence X-ray scattering techniques"

Self-assembling side-chain liquid crystalline diblock copolymers (SCLC-BCs) combine two self-organizing mechanisms in a single molecular system at different length scales: the microphase separation between the two polymer blocks and the LC ordering inside one of them. The tandem effect of these two mechanisms can be used to prepare laterally patterned surfaces on the nanoscopic length scale. Block copolymers that form cylindrical nanodomains have special importance due to the potential applications they can offer. Selectively removing the minor component transforms them into nanoporous media, membranes, lithographic templates, nano channels and scaffold to assemble electronic materials. We used an asymmetric poly(methyl methacrylateb-1H, 1H, 2H, 2H-perfluorodecylacrylate) diblock copolymer to prepare thin films of cylindrical nanodomains normal to a supporting silicon substrate. Here, we use grazing incidence x-ray scattering to obtain information on both the lateral and v ertical ordering nanostructured of these thin films. Specular x-ray reflectivity (XR) measurements were used to probe the electron density profile along the surface normal direction. Modelling the measured reflectivity profile gave about the ordering and layering in the normal direction. Lateral ordering was studied by grazing incidence small angle x-ray scattering (GISAXS) by varying the momentum transfer qy in the lateral plane. The thin films were also characterized using atomic force microscopy (AFM). The combination of the scattering techniques and scanning probe microscopy provides a compelling evidence for their nanostructures.

A5. J.W.Andreasen, M.Jørgensen, F.C.Krebs Danish Polymer Centre, Technical University of Denmark

"The nano-scale structure of bulk heterojunction photovoltaics, investigated by GISAXS"

A bulk heterojunction structure for efficient charge separation in photovoltaics consists of a well dispersed interpenetrating percolation network of donor and acceptor. We demonstrate the application of the GISAXS technique for characterisation and optimisation of such structures, formed by self assembly.

A6. T. Arnold, M.H. Burt, C.L. Nicklin and U.H. Wagner Diamond Light Source

"XENA: a new beamline for GISAXS, GIXD and XRR"

Beamline I07, XENA (X-ray scattering Experiments for Nanostructure Analysis) is a high-resolution surface Xray diffraction beamline presently under construction at Diamond Light Source in the UK, scheduled for completion in 2009. The design of XENA includes the capacity for Grazing Incidence Small Angle X-ray Scattering (GISAXS) and X-ray reflectivity (XRR) techniques in addition to Grazing Incidence X-ray Diffraction (GIXD) and will enable the structure of surfaces and interfaces to be investigated under a wide range of well defined and controllable environments. Further, the collocation of this facility with complementary surface neutron scattering instruments at ISIS will facilitate the combined power of both techniques to be applied to complex surface structures. The beamline will compete well in terms of flux with the ID beamlines at the ESRF up to 10 keV. The use of interchangeable environmental stages (e.g. UHV, electrochemical and high gas pressure) will mean, however, that a greater range of technically demanding systems will be studied. These will include complex alloy semiconductor and oxide surfaces, quasi crystals, solid-solid, gas-solid and liquid solid interfaces, polymer and biological films. The exceptional quality of the X-ray beam delivered from the undulator source will extend the technique in two important ways; through the use of coherent scattering and by the application of microfocus optics. Both will have a major application to the study of more typical, but disordered surfaces.

A7. D. Babonneau, S. Peripolli¹, M.F. Beaufort, J.F. Barbot and J.P. Simon² Lab Métallurgie Physique, UMR6630 CNRS, Univ. de Poitiers, SP2MI, BP 30179, 86962 Futuroscope, France ¹Instituto di Fisica, Universidade Federal do Rio Grande, CP15051, 91501-970 Porto Alegre, Brazil ² SIMPAP, CNRS-UJF-INPGrenoble, BP75, 38402 Saint Martin d'Heres, France "GISAXS studies of nanocavities and defects induced by He and Ne implantation in Si"

The growth and thermal evolution of nanocavities produced by ion implantation into silicon have received considerable attention because of their application as effective impurity gettering sites for heavy metals [1]. The study of the basic mechanisms involved in the formation of cavities and extended interstitial-type defects is also of great scientific interest for fundamental researches of damage buildup in semiconductors [2]. Actually, cavities and associated defects are usually observed by transmission electron microscopy (TEM), so that most of the available data are obtained after annealing, whereby large voids are formed [3]. We will show that grazing incidence small-angle x-ray scattering (GISAXS) may be a powerful tool for the study of the nanocavities and associated defects in the as-implanted state since the technique is sensitive to the electron density modulation obtained in a non-destructive way and averaged over a large amount of material [4, 5]. He⁺ and Ne⁺ have been implanted into (001) and (111) Si wafers at different temperatures ranging from 100 to 600 °C and different fluences from 7.10^{15} to 5.10^{16} cm⁻² while keeping the incident energy at 50 keV. GISAXS experiments have been performed at ESRF on the D2AM beamline collecting the scattered intensity with a 2D CCD camera. The angle of incidence has been monitored from 0.18° to 0.50° in order to investigate the morphology of the cavities and associated defects from the very surface to deeply buried regions. Quantitative analysis of the GISAXS data in the framework of the distorted wave Born approximation show that low temperature implantations (< 300 °C) lead to the formation of spherical nanocavities with size between 1 and 3 nm and heterogeneous depth distribution for both helium and neon implantations (Figs. 1a and 2). Implantations of neon ions at higher temperatures cause growth of the cavities and broadening of the size distribution without modification of their shape. In contrast, a substantial evolution is evidenced for helium implantations at high temperatures, including {111} faceting and formation of {113} extended interstitial-type defects (Figs. 1b and 1c). From GISAXS simulations and complementary TEM observations, we will show that the cavities are characterized by a size-dependent shape anisotropy due to a size-dependent faceting mechanism. Moreover, it will be demonstrated that the method allows to characterize the morphology of the {113} defects with an evolution from rod-like to ribbon-like type as the temperature increases. References

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Figure 1(left): 2D experimental GISAXS patterns of Si (001) implanted at (a) 200 °C, (b) 400 °C, and (c) 550 °C with 5.10^{16} He⁺.cm⁻².

Figure 2 (right): Atomic concentration profile of Ne and He as determined by simulations [6] for 5 x 10^{16} ions.cm⁻² implanted in Si at 50 keV. The variation of the penetration depth of the incident X-ray beam in Si at λ = 0.139 nm is also shown as a function of the grazing angle.

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"WAXS simulations of textured films"

We present an efficient and user friendly computer program for simulating grazing incidence wide angle scattering (GIWAXS). Particular attention is given to textured polycrystalline films, which can often be modeled as a "cylindrical" (2D) powder. The practical use of the program is demonstrated by examples of relevance for molecular electronics.

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"Thin Films of Semifluorinated Liquid Crystalline Side Chain Block Copolymers"

Fluorinated materials are interesting as coatings because of their low surface free energies. One possible approach to increase processability is the incorporation of semifluorinated alkenes as side chains to a polymerbackbone, which is part of a diblock copolymer.[1] The side chains arrange into liquid-crystalline, smectic layers, which increases the structural complexity compared to block copolymers with two amorphous

blocks. GISAXS and grazing incidence diffraction has been applied to investigate the hierarchical structure of a thin film of a diblock copolymer consisting of a polystyrene block and a semifluorinated, liquid-crystalline (LC) block. The focus will be the interplay between the self organization of the side chains into smectic layers and the mesoscopic arrangement of the polystyrene blocks with respect to the LC-block, which is strongly related the surface properties of the films. [1] S. Krishnan, Y.-J. Kwark and C. K. Ober, The Chemical Record, 2004, 4, 315.

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Characterization of Ge islands on Si(100) substrates

Abstract: We present a study of Ge islands formation on Si(100) substrates using grazing-incidence small-angle X-ray scattering (GISAXS). Samples were prepared by a high-vacuum evaporation of a 5nm thick Ge layer on Si(100) substrate held at 200 °C The samples were subsequently annealed at different temperatures for 1h in vacuum, yielding to island formation. A Fortran program IsGISAXS was used for the simulation and analysis of Ge islands. Vertical cut (perpendicular to the surface) of the experimental 2D GISAXS pattern has been fitted using a Guinier approximation. Obtained parameters were used for the simulations. The simulated 2D GISAXS pattern well reproduce experimental data for cylindrically shaped islands.

A11. Ya-Jun Cheng1, Markus Wolkenhauer1, Gina-Gabriela Bumbu1, Stephan V. Roth3, Jochen S. Gutmann1, 2*

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"GISAXS studies on Titania ultrathin films with different morphologies"

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. The lateral structures of the titania ultrathin films are studied by GISAXS.

A12. C. Darko 1, E.-E. Metwalli-Ali 1, I. Botiz 2, G. Reiter 2, D.W. Breiby 3, S.V. Roth 4, C.M. Papadakis 1 1. Physikdepartment E13, Technische Universität München, James-Franck-Str. 1, D-85748 Garching, Germany 2. Institut de Chimie de Surfaces et Interfaces, CNRS, 68057 Mulhouse, France 3. Niels Bohr Institute, University of Copenhagen, Universitetsparken 5, 2100 Copenhagen, Denmark 4. Hasylab at DESY, D-22603 Hamburg, Germany

"Thin films of diblock copolymers having one crystalline block"

The surface morphologies obtained for thin films of semicrystalline diblock copolymers are strongly influenced by the internal structural rearrangement within the films. In thin films of semicrystalline diblock copolymers, the final structure formed depends on the competition between three transitions: the order-disorder transition of the diblock copolymer, the crystallization of the crystallisable block, and the vitrification of the amorphous block. Using low and high molar masses of a lamellar forming poly(styrene-b-ethylene oxide) diblock copolymers, we have studied the interplay between crystallization and mesophase formation within the thin films. Combining grazing-incidence small and wide-angle X-ray scattering (GISAXS) with grazing-incidence X-ray diffraction (GIXD), we obtain information on the inner structures complementary to the surface textures measured by optical and atomic-force microscopy (AFM).

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3. IMFUFA Roskilde University Denmark

"Transient states during vapor treatment of thin, lamellar diblock copolymer films"

For the use of thin, nanostructured block copolymer films, their stability and their response to changes of the environment are of great importance.

We have studied the structural changes in thin poly(styrene-b-butadiene)(PS-PB) diblock copolymer films and their kinetics during vapour treatment. For this purpose, we use in-situ grazing incidence small angle x-ray scattering (GISAXS). A time resolution of 10 sec can be achieved. Films with the initially parallel or perpendicular orientation were investigated, and both non-selective and selective solvent were applied. Complex processes for film swelling and lamellae reorientation were observed during vapour treatment of spin-coated block copolymer films.

A15. P. Dubcek1, B. Pivac1, I. Capan1, S. Bernstorff2, B. Vlahovic3 1R. Boskovic Institute, P.O. Box 180, Zagreb, Croatia 2Sincrotrone Trieste, SS 14, km 163.5, Basovizza (TS), Italy 3NCCU, Durham, NC, USA

"Evolution of nanoparticles in gold implanted glass"

The interest in metal nanocrystals is focused on their nonlinear optical properties, as they are expected to have a large third order nonlinear susceptibility. In order to explore the possibility of tuning the size and shape of metal nanocrystals we implanted gold into fused silica substrate. The implantation energy was 1.1 MeV and doses were of 1, 3, 6 x 10E16 ions/cm^2. All samples were subsequently annealed in air at different temperatures in the range from 900 to 1100°C. The grazing incidence small-angle X-ray scattering (GISAXS) was used to study the morphology and the distribution of gold nanoparticles formed in the substrate. The clastering process is evident already during the implatation itself. Upon annealing, the number of clusters, as well as their size is increased. The clasters are smaller in the vicinity of the surface, where the effective implant concentration is lower, regardless the annealing temperature. At the highest annealing temperature the clustering diverges into two distinct size distributions.

A16. J. Feldkamp, C. Schroer, M. Kuhlmann, S.V. Roth, A. Timmann, R. Döhrmann, M. Dommach, and R. Gehrke Institut für Strukturphysik, TU Dresden HASYLAB at DESY, Hamburg

We combine classical scattering techniques with tomographic approaches to gain real-space information about the sample on a virtual slice in addition to reciprocal space data. The concept is illustrated with experiments done previously on HASYLAB beamline BW4. Possible enhancements of the technique are discussed.

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Tba

A18. K. Heo, K. S. Oh, J. Yoon, K. S. Jin, S. Jin, C. K. Choi, and M. Ree* National Research Lab for Polymer Synthesis & Physics, Department of Chemistry, Pohang Accelerator Laboratory, Center for Integrated Molecular Systems, Polymer Research Institute, and BK21 Program, Pohang University of Science and Technology, Pohang 790-784, Republic of Korea Department of Physics, Cheju National University, Jeju 690-756, Republic of Korea Quantitative

tba

"Characterization of Low-k Dielectric Films with Bistrimethylsilylmethane using GISAXS and X-ray Reflectivity"

non-destructive grazing incidence small-angle X-ray scattering (GISAXS) and specular X-ray reflectivity (XR) analysis with synchrotron radiation sources, along with spectroscopic ellipsometry analysis, were successfully used to characterize a series of low dielectric constant (low-k), nanoporous SiOCH dielectric thin films with nanometer-scale thicknesses prepared by a radio-frequency inductively coupled plasma chemical vapor deposition (ICPCVD) of bistrimethylsilylmethane (BTMSM) precursor and oxygen gas at various flow rate ratios followed by annealing at 25, 200, 300 or 400 °C. These analyses provided important information on the structures and properties of the nanoporous films. The average size of the generated nanopores in each film was 3.07 nm in radius or less, depending on the process conditions. The film electron densities ranged from 414 to 569 nm-3, the refractive indices ranged from 1.434 to 1.512 at 633 nm wavelength, and the porosities ranged from m 16.1% to 38.9%. Collectively, the present findings show that SiOCH thin films of the type reported here are suitable for use as low-k interdielectric layer materials in the fabrication of advanced integrated circuits.

This work was supported by the National Research Lab (NRL) Program (Contract No. 2005-01385) and the Science Research Center (SRC) Program (Center for Integrated Molecular Systems at Postech) of the Korea Science and Engineering Foundation (KOSEF), by the Ministry of Commerce, Industry and Resources (MCIR) and the Ministry of Science and Technology (MOST) (System IC 2010 Project), by the Nuclear R&D Programs of MOST, and by the Korean Ministry of Education (Brain Korea 21 Program).

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"Imprinting well-controlled closed-nanopores in spin-on polymeric dielectric thin films with using six-armed star shape porogens"

Polymethylsilsesquioxane (PMSSQ) has recently received much attention as an alternative to the workhorse dielectrics silicon dioxide (k=3.9-4.3), because of its relatively low dielectric constant (k=2.7), minimal moisture uptake, high thermal stability. Although the k value of PMSSQ is considerably lower than those of silicon dioxide and silicon nitride, it is still much higher than that of air, k = 1.0, which is the lowest value attainable. Hence there has been much interest in incorporating air into dielectric materials to produce porous materials with low k values (≤ 2.5). One approach is the template-curing reaction of PMSSQ precursors in the presence of thermally labile organic polymer porogens, followed by the creation of pores in the resulting dielectric material by sacrificial thermal decomposition of the porogens. However, during the template-curing reaction, the porogen molecules tend to be phase separated, causing aggregation; this phenomena leads to the formation

of larger-sized pores and limits the porosity of the final dielectric thin film. In particular, star-shaped and dendritic porogens with a high number of arms show a strong tendency toward segregation and aggregation. Thus, it is necessary to minimize the aggregation of porogen molecules loaded in the dielectric matrix. In the presence study, we aimed to minimize aggregation of a six-armed porogen in a dielectric matrix by chemical modification of the porogen end-groups. To test the efficacy of the proposed modification, the nanostructures and properties of porous dielectrics prepared using different amounts of the modified porogen were quantitatively characterized. A soluble PMSSQ precursor containing reactive ethoxysily and hydroxysily groups was used as the dielectric material, while a six-armed poly(ε-caprolactone) with and without triethoxysily termination (mPCL6 and PCL6, respectively) were used as thermally labile porogens.

A20. J Hiller, S Rogers, and N Terrill

"GISAXS and micro-GISAXS on the non-crystalline diffraction beamline I22 SAPPHIRES at Diamond Light Source"

I22 SAPPHIRES (Small Angle Photons for Resonant and Elastic Scattering) is the non-crystalline diffraction beamline currently being commissioned at Diamond Light Source, the UK's new third-generation synchrotron. A GISAXS functionality with the main beam as well as a GISAXS capacity on the optional microfocus end station are planned. We present here the primary beamline specifications and the details of the proposed GISAXS development.

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"Temperature dependent GISAXS experiments at BW4"

In the frame work of a long term project at HASYLAB (2005II011), we developed a thermo-stage with a maximum temperature of 100 Celsius has been built at BW4 under the GISAXS geometry. And it is now fully available for the user community. This poster present the design of the thermo-stage and some GISAXS experiments about anealling polymer films carried out by using this thermo-stage at BW4.

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A23. Sangwoo Jin, Jinhwan Yoon, Kyuyoung Heo, Kyeong Sik Jin, Byeongdu Lee, Weontae Oh, Jehan Kim, Kwang-Woo Kim, and Moonhor Ree*

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"In-situ Grazing Incidence X-ray Scattering Studies on the Formation of Nanopores in Dielectric Films with using Four-Armed Star Shape Porogens"

Porous organosilicate materials have recently attracted much interest due to their potential application as low dielectric constant (low-k) interdielectrics. In particular, much effort has been directed towards the development of low-k porous organosilicate dielectric thin films via the templated polycondensation of their soluble precursors in the presence of a thermally-labile, organic polymeric porogen. Pores are subsequently formed in the resulting dielectrics through the sacrificial thermal decomposition of the porogens in the range 350-400°C. However, the tendency of porogens to aggregate in organosilicates has limited the ability to reduce the pore size and porosity of the resulting dielectrics, making them unsuitable for use in advanced integrated circuits (ICs) patterned with small feature sizes. The formation of porous films from an organosilicate precursor polymer/porogen composite is a complex procedure since the matrix precursor readily undergoes crosslinking w hile the porogen undergoes thermal degradation. In order to understand the structure of pores in the porous film, it is necessary to investigate their generation during the porous dielectric film process. In this study, we further present the in-situ GISAXS study of the evolution of nanopores during porous dielectric film formation in a composite film containing an PMSSQ precursor and a star-shaped modified four-armed poly(*c*-caprolactone) (mPCL4) porogen in various compositions. Using a synchrotron X-ray source, in-situ GISAXS measurements were carried out during thermal treatment of the PMSSQ/porogen composite films to 400°C in vacuum, and continued during the subsequent cooling of the resulting porous films. In addition, thermogravimetric analysis (TGA) and TEM measurements were performed. Detailed analyses of the measured two-dimensional (2D) GISAXS data was accomplished using a GISAXS formula derived under the distorted wave Born approximation (DWBA). The following structural characteristics of both the composite films and resulting porous films: miscibility of precursor matrix and porogen, phase separation, pore generation mechanism, pore size and size distribution, fil m electron density, and film porosity, were also determined. The effect on chemical modification of porogen discussed in detail.

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"Sttering Studies of Nanoporus Organosilicate Thin Films Imprinted with Reactive Star Porogens"

In this study, we aimed to minimize aggregation of a star-shaped poly(ε-caprolactone) porogen with four arms in a PMSSQ dielectric matrix by chemical modification of the porogen end groups. A soluble PMSSQ precursor

containing reactive ethoxysilyl and hydroxysilyl groups was used as the dielectric material, while a four-armed poly(ε -caprolactone) with and without triethoxysilyl termination (mPCL4 and PCL4) were used as thermally labile porogens. PMMSQ/porogen blend films were prepared by partial curing at 200 °C for 100min in a vacuum. The thickness of films were ca. 700nm 2D GISAX and 2D TSAXS measurements were performed in a vacuum using Synchrotron X-ray beam.

From our analyses of the GISAXS and TSAXS profiles, we determined the size and size distribution of pores in the porous films. Overall, the pore size and size distribution determined from the GISAXS data were in reasonably good agreement with those obtained from the TSAXS data. For films imprinted with PCL4, the average radius of gyration of the pores increased substantially, from 4.4 to 5.3 to >40nm, as the porogen loading was increased from 10 to 30 wt %. PCL4 tends to undergo severe aggregation in the PMSSQ matrix at loadings greater than 20 wt %, creating large pores in the films. Compared to PCL4, mPCL4 was found to give pores of smaller size and narrower size distribution, with values in the range 4.0-17.1 nm for mPCL4 loading of up to 30 wt %. These results indicate that severe aggregation of mPCL4 is suppressed in the PMSSQ matrix. This suppression of mPCL4 aggregation can be attributed to the triethoxysilyl terminal groups of the porogen, which are analogues of th

e reactive functional groups of the PMSSQ precursor that take part in the curing reaction during the film formation process. Because of the similarity of the two types of groups, mPCL4 is more miscible with the PMSSQ precursor than is PCL4, and furthermore, the triethoxysilyl terminal groups cause the porogen to participate in the curing reaction of the PMSSQ precursor, leading to a significant inhibition of aggregation in the mPCL4/PMSSQ system.

A25. Andreas Keilbach, Ralf Köhn, and Thomas Bein Department of Chemistry and Biochemistry, University of Munich, Munich/Germany

"2D SAXS on PMO Materials confined in Anodic Alumina Membranes"

Periodic mesoporous organosilica (PMO) materials can be synthesized from bis(alkoxysilyl) precursors (RO)3Si R' Si(OR)3 in the presence of surfactants such as tetraalkylammonium halides or nonionic triblock-copolymers. This way it is possible to produce materials with both high loading of organic functional groups, homogeneously distributed throughout a silica matrix, and highly ordered mesoporous structures. Previously PMO materials have been synthesized in the form of powders and thin films. Here we report the synthesis of PMO materials within a porous anodic alumina membrane (AAM) host system, thus combining the advantages of thin films with the orientation and high aspect ratios featured by the anodic alumina channels. The formed mesophases were studied using 2D-SAXS in combination with TEM measurements.

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"Synchrotron Grazing Incidence X-ray Scattering Studies on Structures in Thin Films of a Brush Polymer"

The preparation of self-assembly polymeric materials has received much attention due to the possible application in electronic and biological materials. In particular, polymers consisting of flexible backbone and bristles were known to form side chain crystalline phase and/or side chain liquid crystalline phase, while these polymers with shorted side chains were known to form amorphous phase. So far, however, most of these studies were concerned with bulk material; however, their thin films were rarely studied. In fact, it is very interesting to investigate the behavior of these polymers in thin films because the molecular ordering may be affected by both the substrate-polymer interface and the polymer-air interface; such behaviors may be quite different from the bulk. Furthermore, it is also necessary to understand the structure and structural transition in the thin film onto substrates when the polymers forms side chain crystalline phase at lower temperature and undergoes thermal melting transition behavior at higher temperature. In the present study, poly(oxyethylene)s containing alkyl side groups were synthesized and then their thin films were prepared on silicon substrates. These thin films were investigated with using synchrotron grazing incidence X-ray scattering and differential scanning calorimetry as a function of temperature. The results will be discussed in detail with considering the backbone and bristles as well as their ordering and phase behaviors. This study was supported by the Korea Science & Engineering Foundation (National Research Lab Program: Contract No. 2005-01385) and by the Ministry of Education (BK21 Program). Synchrotron GISAXS measurements were supported by the Ministry of Science and Technology and POSCO.

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A28. D. Korolkov, P. Busch, E. Kentzinger, L. Willner and Th. Brückel Forschungszentrum Jülich GmbH, Institut für Festköperforschung

"GISAXS and GISANS on self-organized diblock-copolymers"

Fabrication of periodic nanoscale structures using self-organizing systems promises to be a simple and low cost method with potential to produce high-density arrays of electronic and magnetic devices. A block-copolymer can self-assemble into microphase separated domains with a spacing of 10-100 nm which strongly depends on molecular weight, segment size, and the strength of interaction between the blocks. In this study self-organization of polystyrene-polybutadiene (dPS-PB) diblock copolymer with various thicknesses and molecular weights was investigated. Structural characterization was done by combining a surface-sensitive technique like atomic force microscope (AFM) with grazing incidence small angle x-ray and neutron scattering.

A32. M. Kriechbaum, M. Steinhart(2), B. Sartori, M. Rappolt, P. Laggner and H. Amenitsch. Inst. of Biophysics and Nanosystems Research, Austrian Academy of Sciences, Graz, Austria (2) Inst. of Macromolecular Chemistry, Czech Academy of Sciences, Prague, Czech Republic

"Non-lamellar phases of solid supported phospholipids studied by high-pressure GISAXD"

Lipid samples were deposited and aligned on a solid support (Si-wafer) and placed in a hydrostatic pressure-cell (up to 2 kbar) for GISAXD measurements at the SAXS-beamline at ELETTRA.

A29. M. Kumpugdee-Vollrath, T. Ngawhirunpat1, P. Opanasopit1

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Determination of structure of pharmaceutical systems by using a synchrotron source

This present study determines different pharmaceutical systems e.g. liposomes, polymer micelles, lipid emulsions and shed snake skins in order to understand the orientation of the structure in nanoscale. The techniques e.g. small angle x-ray scattering (SAXS) and x-ray microscopy from the synchrotron source are suitable for this purpose because this source can produce a highly intense and focus x-ray. Very comprehensive informations up to the nanoscale are the result. However, these modern techniques can be performed only at the large scale facilities e.g. Berlin Synchrotron Source (Bessy), Berlin/Germany, German Synchrotron Source (Desy), Hamburg/Germany. These techniques are until now mostly used in the field of physic, biology and chemistry. This present study shows, however, that these techniques are also useful as complementary techniques for pharmaceutical systems. The results of the study of the crystalline systems e.g. liposomes, lipid emulsion, shed snake skins by the SAXS-technique gave informations about the lipid structure and also the phase transitions as the measurement can be performed by varying the temperatures. Moreover a deeper understanding can be obtained in combination with the microscopic techniques e.g. x-ray microscopy or atomic force microscopy.

A30. Sebastian Lenz1 and Jochen S. Gutmann1,21Max Planck Institute for Polymeric Research, Mainz2 Institute for Physical Chemistry, Johannes Gutenberg University, Mainz

"Phase behaviour and transition of polymer-brushes on Microcantilevers"

Polymer brush coatings are well known for their ability to tailor surface properties in a wide range of applications. In most cases, brushes are used in solution.

Here we show that the micromechanical cantilever (MC) sensor technique is a tool to perform time-resolved physico-chemical investigations of the phase behaviour and phase transition of thin polymeric layers. Complementary to scattering techniques like GISAXS or Neutron reflection for investigating the change of microscopic properties of the thin films, MC sensor technique provides information about changes in the internal pressure of brushes during a phase-transition.

B30. Byeongdu Lee*, Young-hee Park*, Yecheol Rho, Byungcheol Ahn, Moonhor Ree†

"Ultralow-k nanoporous organosilicate dielectric films imprinted with dendritic spheres"

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"GISAXS on alloy nanostructures grown by quasi van der Waals epitaxy"

We have studied the morphology of epitaxial alloy nanostructures using grazing incidence small angle x-ray scattering. The motivation is to study the influence of the long range chemical ordering on the shape and size of CrPt3, FePt and CoPt alloy nanostructures. The nanostructures were grown by co-deposition on the (0001) van der Waals-type surface of WSe2 under UHV conditions at different temperatures. Due to the weak interactions between metal adatoms and the substrate, alloy nanostructures are formed by self-assembly. In spite of the large lattice mismatch(~17%), the nanostructures grow with the (111) plane azimuthally aligned with the WSe2(0001) plane and are approximately stress-free. For deposition up to 100°C the alloy nanostructures adopt the fcc solid solution and above 100°C start to order in the L12 cubic phase for CrPt3 and L10 tetragonal phase for CoPt and FePt[1,2].

The GISAXS experiments were performed on the french CRG-BM02 beamline at the ESRF, using a photon energy of 7keV. Submonolayer deposits of CrPt3, CoPt and CoPt were prepared at 300 and 500°C in an UHV chamber on WSe2 substrates sticked on STM plates. They were transferred using a vacuum valise on BM02 and centered in the GISAXS chamber operating under 10-5 torr. The GISAXS patterns were recorded under an incidence angle of 0.4deg (i.e. slightly below the critical angle of WSe2). Most of them exhibit interference peaks signature of an average separation distance between nanostructures, which increases with increasing the deposition temperature. Preliminary radial analyses of GISAXS intensities are performed in vertical and horizontal regions in the (qy, qz) plane which are centered around the interference peak and the qz-value corresponding to the maximum intensity, respectively. Using the Guinier approximation, estimates of lateral size and height can be extracted showing a significant increase of the lateral size for the CrPt3 nanostructures with the growth temperature, while for CoPt and FePt no significant change is observed. This different behaviour can be attributed to the structure of the LRO ordered phase adopted by the nanostructures, i.e. L10 for CoPt or FePt and L12 for CrPt3. To determine the precise morphology of these nanostructures, simulations of the complete GISAXS patterns using the IsGISAXS program written by R. Lazzari have been performed. The best simulations for the CrPt3 deposits at 500°C are obtained using a truncated tetrahedron for the island shape with the following geometrical parameters: 5nm for the length of the edge of its bottom triangular facet and 1.2 nm for its height, and a paracrystal model to simulate the interference function leading to a separation distance around 17nm. For FePt deposits, interference functions are extracted from STM images and their use in the GISAXS simulations is foreseen.

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"Nanostructured titania nanoparticles by different block copolymers as templates"

Nanostructured inorganic semiconductors as the electron transport component in organic hybrid solar cells are promising candidates for low light solar cells. The effect of titanium oxide (TiO2) nanoparticles on the efficiencies of conjugated polymer photovoltaics has been widely studied. Our group achieved the preparation of TiO2 thin films with variable morphologies in a reproducible way through a simple recipe by using polystyrene

(PS) block polyethyleneoxide (PEO) copolymer as a template [1,2]. Low melting and glass-transition temperatures, and ability to turn into silicon–oxi-carbide (Si-O-C) after being heated to elevated temperatures give polydimethylsiloxane (PDMS)-containing block copolymers unique properties for diverse applications. In this study, our aim is to synthesize ordered self encapsulated Si-O-C/Titania core-shell nanoparticles via using PDMS-b-PEO copolymers. Then heat the block copolymer-titania nanocomposite films to elevated temperatures and analyze t he Titania nanoparticle morphology as well as ordering by using Scanning Electron Microscopy and Microbeam Grazing Incidence Small Angle X-ray Scattering Techniques. Synthesis of the required block-copolymer and their suitability for use in dye sensitized solar cells will be addressed.

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"Structural investigation of casein micelles in thin films"

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 precleaned 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.

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"Cholera Toxin Assault on Lipid Membranes Containing Ganglioside GM1. An X-ray Reflectivity and Grazing Incidence Diffraction Study at the Air-Water Interface"

Cholera toxin is highly efficient in taking over host organisms. To exert its destructive effects on the cell, cholera toxin must bind to and infiltrate the cellular membrane, a specialized and controlled barrier. The mechanism by which cholera toxin crosses the membrane remains unresolved. Using x-ray reflectivity, we have been able to monitor the binding of cholera toxin, show that protein coverage ranged from approximately ½ to 2/3 maximum occupancy, and follow the penetration of the cholera protein into the lipid monolayer at the air-water interface. Protein penetration in the absence of cholera toxin's A subunit at pH=8 may suggest that the B5 pentamer plays a more active role in the membrane penetration mechanism than solely binding cholera toxin to its cell surface receptor. Grazing incidence x-ray diffraction revealed a decrease in the in-plane and out-of-plane order of the model lipid membrane after protein binding with an additional perturbation after protein act ivation. In addition to disrupting the order, cholera toxin binding also caused an increase in tilt of the lipid molecules with a commensurate thinning of the monolayer. Furthermore, the largest perturbation to the monolayer was caused by the full toxin at low pH, supporting the theory of the important role of low endosomal pH in the infection pathway.

B3. J.-F. Moulin(1), P.M. Buschbaum(1), J.Perlich(1), W.Yang(1), M.Abul Kashem(1), S.V. Roth(2), A. Timman(2)

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GISAXS Investigation of flow

We will present a new setup for the GISAXS study of the interface between a flowing liquid and a substrate. This setup allows one to obtain the scattering of the buried interface and to study the influence of flow parameters (nature of the solution, interactions with the substrate, flow speed...) on the interfacial structure.

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"Polymer coated micro mechanical cantilever arrays studied with micro-focus GISAXS"

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 ink-jet-printing is a smart approach. Using the automatic pipetting system, Nano-PlotterTM (GeSiM) the solution for tethering is applied contact less in small droplets directly to the cantilever. Tethering is done either by a "grafting to" or "grafting from" approach. The small geometry of the cantilever (500 to 1000 μ m length, 90 μ m width, 1 to 5 μ m of thickness) rises problems with the standard techniques for characterising the functional layer. So we used the μ -focus option at the GISAXS Line BW4 at the HASY-Lab in Hamburg for detailed studies.

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"Time-resolved in-situ GISAXS: Structural changes of thin diblock copolymer films in solvent vapor"

Block copolymer thin films form spontanously mesoscopically ordered long-range structures which may have a number of applications, e.g. as templates for the preparation of nanoobjects [1]. However, self-organization is associated with domain formation and defects which hamper the use of block copolymer thin films. Vapor treatment constitutes a route to the formation of well-defined, long-range order structures [2]. By means of grazing-incidence small-angle X-ray scattering (GISAXS), normal and lateral inner film structures can be characterized simultaneously [3].

We present here results from 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 [4]. The films were treated with different solvent vapors which vary in their quality and selectivity. GISAXS experiments were performed at BW4 at HASYLAB/DESY and at D-line at CHESS.

We have found that treatment with solvent vapor has a significant effect on the resulting film structure. For instance, treatment of an OB film with cyclohexane, a selective solvent, leads to the development of Bragg rods due to perpendicular lamellae on the time scale of minutes. These Bragg rods persist upon drying, i.e. the lateral structure is stable.

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"Microbeam GISAXS investigation of sol-gel templated nanocomposite films"

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, therefore it is very important to prepare the desired morphology with high reproducibility and homogeneously spread out over areas of cm2. For the preparation of the TiO2 nanocomposite films we combine the amphilic diblock-copolymer PS-b-PEO, which acts as the templating agent, with an inorganic solgel 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 (GISAXS) is performed at the synchrotron beamline BW4 of the DESY HASYLAB in Hamburg. The investigation is complemented by surface and thin film sensitive probes such as atomic force microscopy, x-ray reflectometry and imaging ellipsometry. We acknowledge financial support by the project MU 1487/5-1 and GU771/2-1.

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"Coarsening Dynamics of Au Precipitates in a Thin Fe Film"

Iron films grown on a Au(100) substrate are one of the best known examples for self-surfacting action promoting a flat 2D-growth [1]. During Fe growth gold atoms are segregating to the iron surface. As a result the iron film is immediately covered by a floating Au layer. At elevated temperatures the segregation process is enhanced and Au precipitates are formed within in the Fe film.

The precipitate growth and coarsening can be monitored in situ by changes in the grazing-incidence small-angle X-ray scattering (GISAXS) diffraction pattern. Furthermore, by combining GISAXS with the method of X-ray photon correlation spectroscopy (XPCS) it becomes possible to analyse the dynamics of the coarsening process near the surface [2,3].

Utilizing coherent X-rays time series of fluctuating speckle pattern are recorded. Information about the underlying coarsening mechanism is gained by analyzing the intensity fluctuations for different scattering vectors.

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B10. B. Pivac, I. Capan, P. Dubcek, N. Radic,R. Boskovic Institute, P.O. Box 180, Zagreb, Croatia,S. Bernstorff,Sincrotrone Trieste, SS 14, km 163.5, Basovizza (TS), Italy

"SUBSTRATE TEMPERATURE DEPENDENCE OF Si NANOSTRUCTURES FORMATION IN SiO/SiO2 SUPERLATTICE"

Silicon due to its indirect band gap, has been considered not suitable for optoelectronics. But since the discovery of intense visible light emission in porous Si in the 1990s an extensive research has been devoted to study of Si nanoscaled structures for optoelectronic and photovoltaic applications. Present research on Si nanosize structures is focused on the Si nanocrystals prepared by sputtering of Si rich oxides and SiO2 multilayers on Si substrates.

We present a study on amorphous SiO/SiO2 superlattice formation on Si substrate held at different elevated temperatures. Grazing-incidence small-angle X-ray scattering (GISAXS) and photoluminescence was used to study such samples. From the 2D GISAXS pattern it is possible to determine the shape, size and inter-particle distance. Amorphous SiO/SiO2 superlattices were prepared by magnetron sputtering of 2nm thin films of SiO and SiO2 (10 layers each) from corresponding targets on silicon substrate. Rotation of the Si substrate during evaporation enables homogeneity of films over the whole substrate. After evaporation samples were annealed at 1050 C in different atmospheres. The analysis of the 2D GISAXS pattern has shown that some Si nanocrystals are already present in some samples deposited at elevated temperatures. Using a Guinier approximation, the inter-nanocrystal distance and the thickness of the nanocrystals have been obtained. A long range ordering of nanoparticles deposited at elevated temperatures are observed.

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"GISAXS Studies of Iron on polished MgO(001)"

Grazing-incidence small angle x-ray scattering (GISAXS) studies of iron islands on polished MgO(001) are presented. Measurements were realized at the high-brilliance undulator beamline ID10A at the ESRF (Grenoble). Due to a missing conducting surface, iron layers on MgO thinner than approximately 20 monolayers (ML) cannot be studied by common methods like scanning tunneling microscopy. Hence, the structural and magnetic properties of iron clusters, resulting from the evaporation of 5 ML iron on a polished MgO(001) substrate are examined by the GISAXS method and in addition by conversion electron mössbauer spectroscopy. Volmer-Weber-growth is observed for room temperature deposition giving rise to spherical superparamagnetic islands. Subsequent annealing results in coarsening and change of the particle shape into a hemisphere. In the early stages of annealing cluster diffusion is the primary process, whereas later Ostwald ripening is the dominant coarsening mechanism. The diffusion coefficient is independent on the crystallographic directions in the MgO(001) surface as it is proven by the isotropic island distribution and form.

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"Current status of the microfocus SAXS / WAXS beamline at PETRA III - design and layout"

Starting in 2007, the new high-brilliance third generation synchrotron radiation source PETRA III at HASYLAB / Hamburg will be constructed. PETRA III as a third generation synchrotron sources allows to realize new experimental methods using microand nanofocused x-ray beams. Hence, among the first beamlines to be built at PETRA III is the microfocus small- and wide-angle x-ray scattering beamline μ SAXS/WAXS. This beamline will exploit the excellent photon beam properties of the low emittance source PETRA III to provide micro- and nanofocused beams with ultra-high intensity and resolution in real and reciprocal space. Based on the first concept for the μ SAXS/WAXS beamline proposed in [1], we redefined the layout of the beamline incorporating on-axis compound-refractive optics. The new design foresees dedicated microand nano-focus end-stations being both dedicated to transmission as well as grazing incidence experiments. The beam dimensions range from 100 μ mx7 μ m and 40 μ mx3 μ m for a microfocus to approximately 100nmx100nm for a nanofocus. Optics under consideration for nanofocusing include waveguides [2], Fresnel-zone plates [3], Beryllium compound refractive lenses [4]. Additionally, a 1:1 imaging layout for combining USAXS with a

moderately microfocused beam is currently discussed. The microSAXS/WAXS beamline is planned to exploit one of a high-beta canted 2m undulator pair. The energy range of the beamline is foreseen as 8-25keV. In combination with Si(111) crystals, this demands a very high stability and precise positioning. To suppress higher harmonics, a planar double-mirror with low incidence angle compatible with the large energy range of the beamline will be used. We will present the current layout of the μ SAXS/WAXS beamline and discuss the different micro- and nanofocusing options possible. Furthermore, we will review the implications of this new beamline with respect to in-situ experiments as well as brilliance-demanding novel methods like μ GISAXS [5] and μ SAXS tomography [6].

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B14. S.V. Roth (*), A. Timmann, R. Döhrmann, M. Dommach, M. Kuhlmann, C. Schroer, O. Leupold, B. Lengeler(2), H. Walter (3), P. Müller-Buschbaum (4), R. Gehrke

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"µGISAXS and µSAXS - new high-resolution small-angle options at BW4 / HASYLAB"

The wiggler beamline BW4 at HASYLAB is dedicated to ultra-small-angle x-ray scattering (USAXS) in material science [1]. After ten years of successful operation major upgrading and refurbishment has been performed, starting in 2003 [2]. 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: a) The maximum USAXS resolution - defined by the size of the beam stop - is dmax > 1 μ m at a sample-to-detector distance LSD = 13m. b) In grazing incidence USAXS (GIUSAXS) the resolution has been pushed well beyond dmax > 3 μ m. c) A new SAXS setup using a newly designed piezo-driven portable slit is available. This allows to optimize intensity and resolution for LSD • 4m. d) A new microfocus of 30 μ mx17 μ m can be achieved. This new option allows for scanning applications like μ SAXS [3] and μ GISAXS with the sample size reduced by two orders of magnitude. Fig. 2 shows a dry rat's tail collagen pattern obtained with the microfocus set-up. In detail, we will discuss μ GISAXS combinatorial investigations on nanocomposite thin films and their relation to optical properties.

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B15. A. S. H. Rozatian (1, 3), P. Vahdani (1), S. E. Nourian (2), C. H. Marrows (4), T. P. A. Hase (3), B. K. Tanner (3)

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"Interface Properties of Co/Pd Multilayers Studied by Grazing Incidence X-ray Scattering Method"

Co/Pd multilayers with Co thickness less than 8 Å show Perpendicular Magnetic Anisotropy (PMA) and have been extensively investigated as candidates for high density perpendicular recording media. In this paper structural properties of a series of sputtered Co/Pd multilayer thin films have been investigated. High-Angle X-Ray Diffraction (HAXRD) measurements using a Cukα laboratory x-ray source indicate a modulated structure which is strongly textured grown along (111) direction. The relation between intensity and the number of bilayers for different peaks of HAXRD spectrum has been investigated. Interface morphology has also been

studied from Grazing Incidence Specular and Off-specular x-ray measurements using synchrotron radiation and has been compared to the HAXRD results.

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"Self assembly of iron oxide nanoparticles as observed by time resolved grazing incidence small angle X ray scattering"

The evaporation of colloidal nanoparticle solutions is known to produce ordered monolayers of nanoparticles. The in situ time resolved grazing incidence small angle x ray scattering was employed to study the self assembling of iron oxide nanoparticles from colloidal solution. The colloidal solution was dropped onto the silicon substrate and non coplanar x ray scattering was recorded as a function of time with the resolution up to 100 ms. The x ray scattering contributions from the evaporating drop volume and surface as well as from the substrate surface were monitored and identified. The x ray scattering from the drop surface for the distances as close as 80 um to the substrate surface. The presented measurements are pointing to the vicinity of the three-phase drop contact line as a region of nanoparticle self assembling.

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"In-situ investigation of the interfaces of a block copolymer solution under shear stress with µ-focus GISAXS"

Block-copolymers are interesting for their ability to self organize in various colloids. In dilute solution they form micelles, cylindrical micelles and vesicles. At higher concentrations they form ordered structures like face-centred-cubic lattices (FCC) or hexagonal packed cylinders (HEX). The length scales of these colloids and structures range from about 5 nm up to several hundred nanometres. Hence such structures are well suited for investigations using small-angle X-ray scattering (SAXS).

The experiments were performed the beamline BW4 at HASYLAB, Hamburg [1] using the micro focus setup. We present the results of the investigation of a 13 wt.% solution of a poly-(isoprene-block-ethylene oxide) in water forming a FCC lattice. The block degrees of polymerization of the polyisoprene and the polyethylene oxide were 55 and 170, respectively.[2] The shear stress was applied by a stress-controlled Bohlin CVO rheometer in a plate-plate-geometry with a diameter of 20 mm and a gap of 1 mm. We investigated the interface layer of the solution with the rotor of the shear geometry. We found a change of the layer thickness of the metal-solution interface under the application of shear. The thickness under shear is independent of the shear rate. References:

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"Fabrication and spectroscopy of nano- and microcrystalline Y3TaO7 and Y3NbO7 activated with Er or Er and Yb"

Classical synthesis of Y3MO7 is based on a solid state reaction and is typically performed at 1600°C for 40 hours or 1400°C for two weeks. We elaborated an alternative method of fabrication of Y3MO7, which is based on a procedure originally described by Brixner for YTaO4. It relies on heating a stoichiometric mixture of yttrium (part of which is replaced with Er, as activator) and niobium oxides with appropriate amount of Li2SO4,

acting as a flux. Using this process of synthesis we prepared the crystallographically pure cubic Y3MO7:Er heating mixture of Y2O3, Er2O3 and Ta2O5 and the Li2SO4 flux at 1300°C for 5 hours. We have fabricated a series of powders of cubic Y3MO7:x%Er (x=0.1-15) and Y3NbO7:0.5%Er, x%Yb (x=5; 10%) using the fluxaided route . Irradiation of the powders with 980 nm photons from diode laser produces visible luminescence consisting of basically two bands located around 550 nm (green) and 660 nm (red). The ratio of intensities of the two components varies systematically with the dopant content. At the lowest concentration of Er the emitted light is located mostly in the green region, while with increasing content of the dopant still more and more light is emitted in the red part of spectrum and finally the red band prevails over the green one significantly. Thus varying the Er concentration it is possible to adjust the up-converted emission color. In regular photoluminescence excited in UV the 660 nm emission intensity also increases with Er content but it never surpasses the green luminescence. We also developed a synthesis procedure of M3TaO7:Er (M=Y, Lu) which is based on Pechini technique. It allows us to obtain both micro- and nanosized phosphors. We prepared powders with dopant concentration varying between 0.1-25%. Luminescence after irradiation with 980 nm diode laser also consist two bands. Their intensities ratio varies with the dopant content - for microsized phosphor emitted light changes from bluish green for 0.1% to orange for 15% and for 25% it becomes yellowish green. For nanosized phosphor light emission color appears in different shades of green - from bluish green for low contents of Er to vellowish green for high concentrations of the dopant. Our research proved that it is possible to adjust the up-converted luminescence color from the investigated materials both varying Er concentration and the crystallites size.

B19. Jin Wang Argonne National Laboratory, 9700 S. Cass Ave., Argonne US

Complex nanocomposites are believed to be associated with novel electronic, magnetic and photonic properties of organic and inorganic components. In these nanocomposite systems, although highly ordered structures can often form in a self-assembled fashion, the formation of the structures can be extremely dynamic, far from commonly believed near-equilibrium conditions even at the end of the ordering processes. Therefore, a controlled self-assembling of the nanostructure has to be guided by a thorough understanding of ordering kinetics and nanoparticle dynamics in the complex matrices. For probing the systems involving dynamical structure of surfaces and buried interfaces, many x-ray surface and interfacial characterization techniques provides a unique scientific opportunity to study the principle of formation of ordered nanostructures. As an increasingly important structural-characterization technique, grazin g-incidence small-angle scattering (GISAXS) finds vast applications in nanostructures and nanocomposites at surfaces and interfaces for in situ and real-time studies because of its probing q-range $(10^{-3} - 1 \text{ nm}^{-1})$ and temporal resolution $(10^{-3} - 1 \text{ s})$. At the APS, we pioneered in using GISAXS techniques under thin-film waveguide-based resonance conditions to study nanoparticle/polymer nanocomposites and the kinematics of ordered nanoparticle formation at air/liquid interfaces. To meet the strong demand from the nanoscience community, a dedicated GISAXS beamline has been designed and constructed as a part of the 8-ID-E beamline at the APS. Taking advantages of x-ray beam from an undulator, this beamline is designed with both simplicity and flexibility in mind to achieve high resolutions in both reciprocal and real spaces as well as high temporal resolution in measurement. The samples can be situated in an integrated vacuum chamber on a high-precision heating and cooling stage. The sample chamber can also be isolated from the beamline to allow solvent flows and to accommodating other mechanical systems such as in situ dip-coating devices. This new and dedicated GISAXS beamline has allowed structural and dynamic characterization of not only nanoparticles of vast varieties but also organic/organic nanocomposites that possess only weak scattering contrast. This new capability presents a unique research opportunity in softcondensed matter research field. A list of new and exciting research programs has been built around the x-ray measurements such as 1) Kinetics of block copolymer thin films under thermal and/or solvent annealing, methods to obtain mesoscaled ordered nanostructures using the phase properties of the polymer blocks, 2) Solgel processes to form highly-ordered nanostructures, effect of the solvent conditions and characteristics, 3) Formation of organic/inorganic nanocomposite, for example, using organic materials as the templates for inorganic nanocyrstals to form superlattices, 4) Preparation and characterization of quantum dots, 5) Dynamics of surfaces, interfaces and nanoparticles in ultrathin films.

B20. Weinan Wang

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B21. M. Wojtowicz, and E. Zych Faculty of Chemistry University of Wroclaw 14 F. Joliot-Curie Street, 50-383 Wroclaw Poland

"Host-related luminescence from nanocrystalline Lu2O3"

Nanocrystalline Lu2O3 powders were prepared with crystallites sizes 6 nm, 30 nm and >100 nm. At least three different emissions were observed upon excitation into the host lattice with 205 nm photons. The emissions were located at 255, 317, 340 and 450 nm. Each of the luminescence bands showed different behavior with temperature as well as with crystallite size. In the presentation the origin of the emissions will be discussed. The most sensitive to grain size and to temperature is the highest energy luminescence at 255 nm. For 6 nm crystallites it disappears below 30 K. However, for larger crystallites trace of this emission may be observed even at 50 K.

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B23. Minhao Yan, and Alain Gibaud Laboratoire de Physique de l'Etat Condensé, UMR CNRS 6087, Université du Maine, 72085 Le Mans Cedex 09, France.

"On the intersection of Grating Truncation Rods with the Ewald sphere studied by GISAXS"

In this work a grating made of lines having a height of 55nm and a period of 450nm has been characterized by Grazing Incidence Small Angle X-ray Scattering (GISAXS). The GISAXS patterns are characterized by a series of spots corresponding to the intersection of the Ewald sphere with the Grating Truncation Rods (GTR). When the grating lines are almost parallel to the direct beam, it is found that the location of these spots is very sensitive to any change in the azimuthal angle. The precise location of the intersection of the GTRs with the Ewald sphere is calculated for any azimuthal angle. In addition, the singular behaviour exhibited by the width of the spots in the qz direction is interpreted.

B24. Jinhwan Yoon1, Seung-Yun Yang2, Byeongdu Lee3, Won-Chul Joo2, Kyuyoung Heo1, Jin Kon Kim2, Moonhor Ree1,*

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" Nondestructive, Quantitative Synchrotron Grazing Incidence X-ray Scattering Analysis of Cylindrical Nanostructure in Supported Thin Films"

Block copolymer thin films with well defined nanostructures have recently received considerable attention for their potential nano-fabrication applications. In these applications, controlling the morphology of the block copolymer thin film, particularly the orientation and ordering of the phase-separated microdomain, is essential. For characterizing the structures and orientation, microscopy tools such as transmission electron microscopy, scanning electron microscopy and atomic force microscopy are commonly used. With these tools, images such as those that show local structures near the surface have been obtained, thus enabling discussion of the underlying physics. From the viewpoint of fabrication, this approach is often sufficient, but from the scientific point of view, X-ray scattering measurements are required because only they provide information on a larger scale at high resolution. In particular, grazing incidence X-ray scattering (GIXS) has emerged as a powerful techn ique for characterizing internal structure of thin film. The X-ray beam impinges at a grazing angle onto the sample slightly above the critical angle, so that the film is still fully penetrated by X-ray. Analytical solutions of GIXS patterns based on the distorted wave Born approximation have been developed to describe the complicated reflection and refraction effects, which are not found in conventional transmission X-ray scattering. Recently we derived a GIXS formula under the DWBA for analysis of the structures in thin films deposited on substrates. Using the derived GIXS formula, the GIXS patterns obtained for polystyrene-b-polyisoprene diblock copolymer thin films with various morphologies (hexagonal, hexagonal perforated layer, and gyroid structure)

deposited on silicon substrate were characterized quantitatively. A representative GIXS pattern and its analyzed one are shown in Figure 1. Here, we attempted the quantitative analysis of the two-dimensional (2D) GIXS patterns of polystyrene-b-polymethylmethacrylate (PS-b-PMMA) diblock copolymer films deposited on silicon substrates. The analysis of the GIXS patterns with using the GIXS theory was successfully carried out, and found that PS-b-PMMA thin films deposited on silicon substrates reveal cylinder microdomains perfectly oriented normal to the substrate surface.

B25. Jinhwan Yoon, Sangwoo Jin, Kyuyoung Heo, Kyeong Sik Jin, Seung Chul Choi, Ye Cheol Rho, Moonhor Ree1*

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" Quantitative Analysis of Molecularly Stacked Layer Structure on Substrate by Synchrotron Grazing Incidence X-ray Scattering "

To obtain nanostructures of paracrystal lattice in large scale, transmission neutron and X-ray scattering (TNS and TXS) have been also widely used. However, these scattering techniques are not applicable for nanostructured thin films because of their low sensitivity and resolution, which are attributed to low scattering volumes. Thus, it is utmost needed to develop non-destructive method to analyze quantitatively nano-structures in thin film on a substrate. Here, we introduce a grazing incidence X-ray scattering (GIXS) technique which allows us to obtain highly intense scattering pattern with high statistical significance even for films with tens of nanometer. However, GIXS needs intensive analysis because of the complicate scattering nature due to reflection and refraction effects, which are not found in conventional TXS and TNS. In this study, we derived the GIXS equation with DWBA for an oriented layer structure and compare with experimental data. We consider a paracrystalline system for layer ordering, and rotation matrix to determine orientation and its distribution of lattice. All of these calculations were performed under consideration of GIXS geometry, since q-space is distorted in GIXS by refraction and reflection effects. Now, we apply the GIXS equation for a thin film with layer paracrystal lattice described above to experimetal GIXS data from S-docosanylcystein thin film. Since S-docosanylcystein has long alkyl chain on Sulfur site of cystein, this molecule are well ordered to layer structure on thin film by hydrophobic interaction of alkyl chain.

In conclusion, we derived the GIXS equation with DWBA for an oriented layer structure and compare with experimental data. In this work, we consider a paracrystalline system for layer ordering, and rotation matrix R to determine orientation and its distribution of lattice. All of these calculations were performed under consideration of GIXS geometry, since q-space is distorted in GIXS by refraction and reflection effects.

B26. Lukasz Zaton and Eugeniusz Zych

"Fabrication of Nanometric Lu2O3:Eu Powders for Digital Medical Imaging with X-Rays"

We shall present a method of making nanocrystalline Lu2O3:Eu powders using homogeneous precipitation with urea from water-toluene phase obtained with the help of a surfactant. The fabricated powders were subjected to various

heat-treatments. XRD, IR, UV-Vis absorption and luminescence spectroscopy of the products will be presented and analyzed. Also analysis of an undoped powder luminescence recorded with synchrotron radiation at various temperatures down to 10 K will be given.

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B28. Aleksander Zych University of Wroclaw, 14 F. Joliot-Curie, Wroclaw PL

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B29. Aleksander Zych, and Eugeniusz ZychFaculty of Chemistry, University of Wroclaw14. F. Jolitot-Curie Street, 50-383 Wroclaw, Poland

"Fabrication and structural and spectroscopic properties of Y2SiO5:Ce powder phosphors"

We shall present new fabrication technique of Y2SiO5:Ce microcrytalline powder phosphors and their structural and spectroscopic properties. We will show that this phosphor may be effectively produced using Li2SO4 flux at 1300 C. We will present SEM and TEM images and results of EDS analysis. Finally we shall present emisison and excitation spectra proving that the powder has interesting spectral characteristics. Unfortuanetly, the flux used promote significant particles growth leading to the average size of the grains in the order of 10 micrometers.

Acknowledgment & Industrial presentations

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BW4 Team HASYLAB Staff DESY-IT

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- 1.4404, 1.4435, copper, bronze Special materials: Inconel 600 / 625, Monel 400,
- Incoloy 800 / 825, Hastelloy C4 / C-276, titanium Comprehensive advice provided in all project phases



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CERN, Europe's largest nuclear research centre cent, curdpe's largest nuclear research centre near Geneva and the German Electron Synchro-tron DESY, one of the world's leading centres for research on particle accelerators based in Hamburg, have been key cooperation partners for product development at SKODOCK for many years.

Modern quality assurance system

The high demands on metal bellows call for a comprehensive quality assurance system that is implemented by SKODOCK to exemplary effect. The company have this in black and white, being documented by DIN EN ISO 9001:2000 certi-fication as well as by international audit and certification organisations. All welding approvals have been updated. If wished, acceptanc and testing can also be performed by SKODOCK works inspectors.





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ie hervorragende Flexibilität macht SKODOCK Metallbälge zum international bewährten Kon-Metailoage zum international bewarnten kon-struktionseltenent. Als Druck (Weg-oder Tempe-ratur / Wegwandler in der Mess- und Regelungs-technik sind sie ebenso gefragt wie als wartungs-freie hermetische Abdichtung von Ventlispinden. Sie sorgen für winkelfehlerfrei übertragende Kupplung rotierender Wellen oder dienen als nurbewardliche Jehrwerzweitkonen in Anen Kupplung rötterender Weilen oder dienen als raumbewegliche Leitungsverbindung im Appa-raten und Aggregaten sowie als fernsteuerbares, pneumatisches oder hydraulisches Betätigungse-lement. Als hermetische, bewegliche Abdichtung von Durchführungen zu Räumen mit z.B. radioak-tiven etwenbescheiden der ihne herbenen. tiver Atmosphäre oder im Hochvakuum.

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ten Projektplanungen werden die Kunden dabei

Das größte europäische Kernforschungszentrum CERN bei Genf und das Deutsche Elektronen-Synchrotron DESY, eines der weltweit führenden Zentren für die Forschung an Teilchenbeschleu-nigern in Hamburg, sind langjährige und wichtige Kooperationspartn wicklung bei SKODOCK. artner für die Produktent-

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Die hohen Anforderungen an die Metallbälge machen ein umfassendes Qualitätssicherungs-system notwendig, das von SKOOCK vorbild-lich ungesetzt wird. Die Zertifizierung nach DIN EN ISO 9001:2000 und durch internationale Klassifikationsgesellschaften dokumentiert das schwarz auf weiß. Alle Schweißzulassungen sind neu erteilt worden. Auf Wunsch können SKODOCK Werkssachverständige auch Abnah-men und Prüfungen vornehmen.

SKODOCK metal bellows are an extremely flex-ible machine part. Designers take advantage of their wide range of possible applications in many different sectors of modern tech-



List of Registrants

05 May 2007, 13:55

GISAXS – an advanced scattering method

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