Emerging Themes in Analysis of Grazing Incidence Small-Angle Scattering Data

Programme and Book of Abstracts

Cosener's House, Abingdon
1st and 2nd July 2013

Table of Contents:

Emerging Themes in Analysis of Grazing Incidence Small Angle Scattering Data

1-2 July 2013 @ Coseners House Hotel, Abingdon

Programme

Monday 1st July:

11:30	Registration and Lunch
13:00	**Welcome**
	Session 1 (Chair: C. Nicklin) – *Sponsored by Rigaku*
13:10	Modelling Grazing Incidence Small Angle X-ray and Neutron Scattering Data (P. Müller-Buschbaum)
13:45	Born Again: Simulating X-ray and Neutron Scattering at Grazing Incidence (G. Pospelov)
14:10	HipGISANS: A Massively Parallel Code for GISAXS Simulation (A. Hexemer)
14:35	Break
	Session 2 (Chair: R. Dalgliesh)
14:45	A Dynamical Theory Applied to Grazing Incidence Neutron Reflection from Periodic Structures (R. Pynn)
15:20	Dewetting of Confined Diblock Copolymer Films Studied by SERGIS (A. A. van Well)
15:45	Coffee break
	Session 3 (Chair: P. Staniec)
16:10	Structure, Morphology and Organization of Nanoparticle During their MBE or CVD Growth by *in situ* GISAXS and GIXD (G. Renaud)
16:45	Observing Thin Film Process Technology *In-Situ* – the Case of Sputter Deposition (S. V. Roth)
17:10	Multi-Lamellar and Non Lamellar Lipid-Aqueous Structures at Interfaces (T. Nylander)
17:35	Break to Rearrange Room for Posters
18:00	**Poster Session with pre-dinner drinks – *Sponsored by SAXSLAB***
19.00	**Dinner – *Sponsored by Dectris***

Tuesday 2nd July:

	Session 4 (Chair: N. Terrill) – *Sponsored by the IOP/RSC Neutron Scattering Group*
09:15	Dynamic GISAXS with Chemical Sensitivity – an Update (R. Winter)
09:50	Grazing Incidence Scattering as a Probe to Determine Structure in Organic Photovoltaic Materials (A. Dunbar)
10:25	Analysis of X-ray Photon Correlation Spectroscopy Data Acquired in Grazing Incidence Small Angle Scattering Geometry (O. Bikondoa)
10:50	Coffee break
	Session 5 (Chair: S. Rogers)
11:15	Mesostructured Self Assembled Polymer-Surfactant Films (K. Edler)
11:45	Directly Programmable Data Analysis Kit (DPDAK) for Online Analysis of High Throughput 2D Scattering Data (G. Benecke)
12:10	Upgrade to the I22 Scattering Beamline to Perform GI-SAXS Experiments (P. A. Staniec)
12:35	Lunch
13:35	Discussion/Breakout Session
14:15	Summary and Closing Remarks
14:30	Close

Abstracts: Invited Presentations

Modeling grazing incidence small angle x-ray and neutron scattering data

Peter Müller-Buschbaum

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Grazing incidence small-angle X-ray scattering (GISAXS) and grazing incidence small-angle neutron scattering (GISANS) emerged to a versatile and frequently used analysis techniques in the field of micro- and nano-structured thin films and surfaces, in particular for soft matter systems [1, 2]. Both are used for the characterization of micro- and nano-scale density correlations and shape analysis of objects at surfaces or at buried interfaces for various classes of materials. As a result, GISAXS and GISANS provide an excellent complement to more conventional nano-scale structural probes such as atomic force microscopy (AFM) and transmission electron microscopy (TEM) [3, 4].

After a short introduction, general principles of GISAXS / GISANS are explained and illustrated with simulations of two dimensional scattering patterns [5]: The scattering geometry, scattering from rough and patterned surfaces and the interplay of object form factor and interference function. Aspects of the interference function and object form factor are deepened by explaining aspects of object shape, object polydispersity and object size. Moreover, layered systems and possible simplifications are discussed.

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A Dynamical Theory Applied to Grazing Incidence Neutron Reflection from Periodic Structures

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As part of an effort to develop a new technique called Spin Echo Resolved Grazing Incidence Scattering (SERGIS) for measuring GISANS, we have carried out a series of measurements of diffraction gratings with various surface profiles. To interpret the data, we have developed a dynamical theory and compared it with other approximations. The dynamical theory provides an accurate description of results obtained with all of the known gratings on which it has been tested. The code that implements the theory is stable to very high orders of Bragg reflection. Comparisons with the Phase Object Approximation and the Distorted Wave Born Approximation illuminate the conditions under which each of these theories can be trusted to give reliable results.

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Structure, morphology and organization of nanoparticles during their MBE or CVD growth by *in situ* GISAXS and GIXD

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Nanoparticles often have new physical or chemical properties (thermodynamic, electronic, chemical, optical, catalytic, magnetic or simply mechanical) because of their small size and/or their particular shape and/or their specific structure (crystallographic structure, strain, composition, defect, surface structure…) at the atomic scale. These structural properties are themselves often determined by the elaboration processes, and sometimes by the structure of the substrate they are grown on. It is thus of utmost importance to investigate the structural properties – structure, morphology and organization – of nanoparticles during their elaboration, in view of understanding their structure/properties relationships.

For that sake, we have developed X-ray techniques to monitor the structures of surfaces/interfaces/nanoparticles *in situ*, in ultra-high vacuum, during the substrate preparation and subsequent growth of nanoparticles on it with the two major techniques that are Molecular Beam Epitaxy (MBE) and Chemical Vapor Deposition. The experimental setup is installed on a synchrotron beamline (CRG/Interfaces, BM32) of the European high energy synchrotron ring, ESRF, in Grenoble. The techniques used are Grazing Incidence X-Ray Scattering/Diffraction (GIXS/GIXD); Surface X-Ray Diffraction (SXRD); Grazing Incidence Small Angle X-Ray Scattering (GISAXS) and X-Ray Reflectivity (XRR), which are all combined with anomalous scattering when needed to assess the composition of the nanoparticles.

In this talk, the UHV/CVD X-Ray chamber and synchrotron setup will be presented, and the principles of the different techniques briefly recalled. A few theoretical considerations concerning GISAXS on organized assemblies of nanoparticles will also be presented. These introductory notions will be followed by several examples of combined structural/morphology studies during the growth of very different systems. The first type of growth will be the Volmer-Weber (3D) growth of metals on oxide surfaces, with emphasis on the growth of Pd, Pt and Ag on MgO(001) and that of Au on TiO_2(110). The second illustration will concern the organized growth of magnetic metallic nanodots on different substrates; either patterned by a surface reconstruction (Co on Au(111), using anomalous GISAXS) or by step arrays and surface reconstruction (Co on Au(677)) or by an underlying dislocation network (Co/Ag/MgO(001) and Ni/CoO/Ag(001)), or by faceting of the substrate (Co on W(111)). The third example will deal with the MBE growth of GeSi islands on Si(001) substrates, as investigated by GISAXS but also Grazing Incidence Multiple Anomalous Diffraction.

Last but not least, recent results recorded during the *in situ* growth of Si and Ge nanowires using CVD, according to the Vapour-Liquid-Solid process will be presented. The evidence for substrate-induced supercooling of the eutectic AuSi catalytic droplets will be discussed, followed by a GISAXS study of the dewetting of Au nanoparticles on Si(111), which is precursory to the growth of semiconductor nanowires .

The care that should be taken when using grazing incidence because of multiple scattering effect will be recalled.

The possibility of combining GIXD and GISAXS *in operando*, to monitor catalytic reactions and the associated nanoparticle modifications, will finally be presented briefly.

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Dynamic GISAXS with chemical sensitivity - an update

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GISAXS is a technique which usually requires stable beam conditions and a sample surface which remains stationary during an experiment to ensure that the grazing angle is constant. This is a rather severe limitation when we want to study processes materials are undergoing rather than just the end result of such processes. Using the calcination of a zirconia gel film on a silicon wafer as an example, we have derived a procedure which enables us to monitor and correct for changes in the sample surface and the grazing angle during a GISAXS experiment with in-situ heating. This involves tracing the sample horizon as a function of time and taking lateral cross-sections at equivalent positions in successive frames (cf. Fig.).
Recently, we have developed a fluorescence correction technique ("absorption-contrast SAXS") in transmission geometry which allows us to obtain scattering data with chemical sensitivity by comparing patterns taken either side of an absorption edge. Unlike anomalous SAXS, this provides chemical contrast at acquisition rates which are useful for studies of dynamic systems. While showing first results from our transmission SAXS experiments around the Zr-*K* edge, we will discuss how we intend to apply this technique in grazing incidence - an experiment we are planning to carry out on beamline I22 later this summer.

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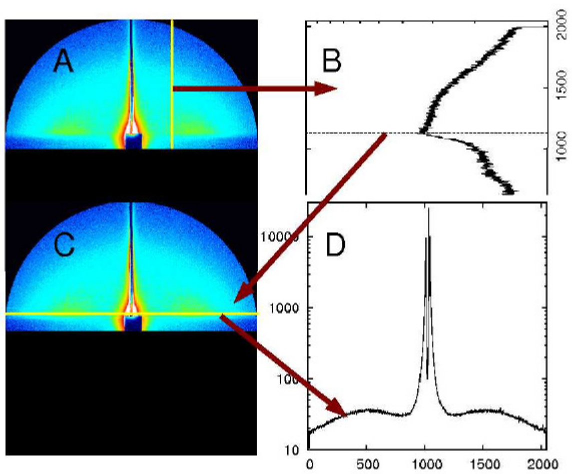

Fig.: Dynamic GISAXS correction procedure. For each frame, the position of the sample horizon is established and a lateral cross-section is taken a fixed distance from the horizon.

Grazing Incidence Scattering as a probe to determine structure in Organic Photovoltaic Materials

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Neutron spin echo resolved grazing incidence scattering (SERGIS) and grazing incidence wide angle X-ray scattering (GIWAXS) have both been used to determine structure in thin films of organic photovoltaic (OPV) active layer materials. The former was used to probe crystallites of [6,6]-phenyl-C61-butyric acid methyl ester (PCBM) produced by extensive thermal annealing of a poly(3-hexylthiophene-2,5-diyl)(P3HT):PCBM organic photovoltaic layer. The latter has been used *in situ* during the film casting process to determine the extent to which the thin films crystallise and indicate the order in which the components of the blends precipitate out of solution.

SERGIS is a relatively new technique and interpretation of the results gained from SERGIS experiments is an area of ongoing development[1]. Upon annealing a thin film of a P3HT:PCBM blend, PCBM crystallites grow on the sample surface. The presence of these crystallites corresponds to the occurrence of a strong SERGIS signal (see figure 1) which is superimposed on the specular reflection from the sample. Features in the data can be correlated with length scales of the crystallites determined using atomic force microscopy and indicate that in such cases the SERGIS signal may be interpreted as a form of small angle neutron scattering.

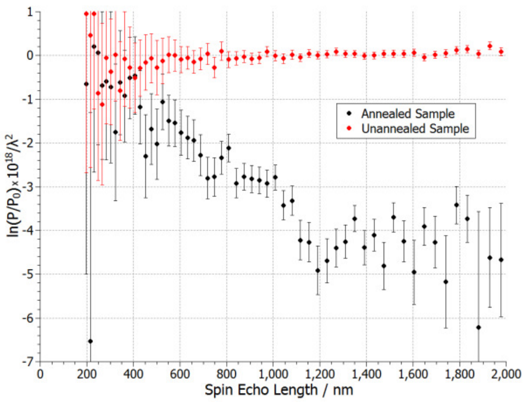

Figure 1: SERGIS data for the annealed and unannealed P3HT:PCBM samples showing distinct depolarization and a plateau starting at approximately 1200 nm in the annealed sample and an effective zero depolarization in the unannealed sample. Reprinted with permission from Appl. Phys. Lett. 102, 073111 (2013); http://dx.doi.org/10.1063/1.4793513. Copyright 2013, AIP Publishing LLC.

In situ GIWAXS studies (in conjunction with simultaneous spectroscopic ellipsometry) conducted during the casting process of similar OPV materials have enabled the evolution of film structure formation to be observed. A number of distinct processes that occur during film formation have been identified, including the growth of nano-scale fullerene aggregates and the onset of Bragg scatter from crystallisation of the conjugated polymers[2,3]. Our observations provide insight into the development of structure in polymer:fullerene blends for OPV applications and potentially assist the future optimisation of this category of materials.

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MESOSTRUCTURED SELF-ASSEMBLED POLYMER-SURFACTANT FILMS

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Solutions of polyelectrolytes with surfactants show a variety of adsorption behaviours at interfaces. We have exploited this phenomenon to create novel solid polyelectrolyte/surfactant films at the air-solution interface, from solutions where the polyelectrolyte is essentially uncharged. In these systems, a self-supporting hydrogel film up to 4 microns thick forms spontaneously at the solution surface. The films contain highly ordered 2D or 3D micellar mesostructures and grow in a few minutes covering the entire interface. Our initial investigations focused on films made from polyethylenimine (PEI) and cetyltrimethylammonium bromide (CTAB) and have been extended to other surfactants with a range of molecular structures, influencing film mesostructure and thickness. More recently we have also prepared films with similar structures to those of films grown at the air-solution interface by spray coating. We are developing these systems for potential applications as a system for encapsulation/release and as a support for sensor species. In this presentation I will describe our grazing incidence diffraction investigations of polymer-surfactant films films aiming to improve our control of nanostructure and the biocompatibility of our materials, and their use as templates for other materials.

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Abstracts: Oral Presentations

BornAgain: simulating X–ray and neutron scattering at grazing incidence

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BornAgain [1] is a multi–platform open–source project that aims at supporting scientists in the analysis and fitting of their GISAS data, both for synchrotron (GISAXS) and neutron (GISANS) facilities. The name of the software, BornAgain, indicates the central role of the distorted-wave Born approximation in the physical description of the scattering process. The software provides a generic framework for modeling multilayer samples with smooth or rough interfaces and with various types of embedded nanoparticles. In this way, it reproduces and enhances the functionality of the present reference software, IsGISAXS by Rémi Lazzari [2], and lays a solid base for future extensions in response to specific user needs. To meet the growing demand for GISAS simulation of more complex structured materials, BornAgain has extended the IsGISAXS program's functionality by removing the restrictions on the number of layers and particles, by providing diffuse reflection from rough layer interfaces and by adding particles with inner structure (see Fig.1 for an example of this last feature). Future extensions will include coverage of polarized GISANS for the investigation of magnetic domains.

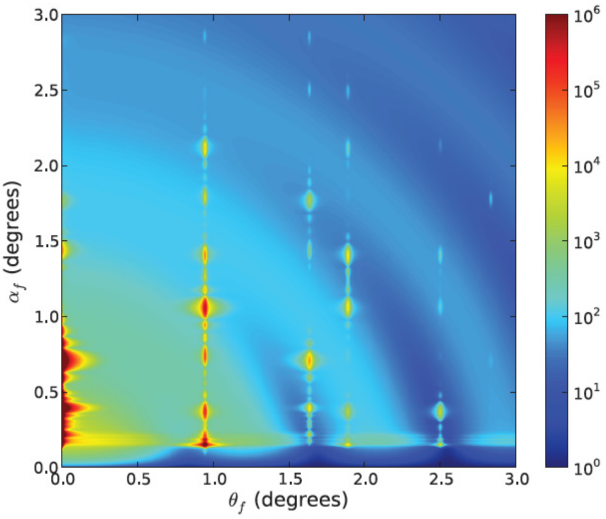

Figure 1: Simulation of grazing incidence scattering intensity for cylindrical mesocrystal islands on a substrate. The mesocrystals consist of an FCC structured self–assembly of spherical nanoparticles.

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HipGISAXS: A Massively Parallel Code for GISAXS Simulation

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Grazing Incidence Small-Angle Scattering (GISAXS) is a valuable experimental technique in probing nanostructures of relevance to polymer science. New high-performance computing algorithms, codes, and software tools have been implemented to analyze GISAXS images generated at synchrotron light sources. We have developed flexible massively parallel GISAXS simulation software ``HipGISAXS'' based on the Distorted Wave Born Approximation (DWBA). The software computes the diffraction pattern for any given superposition of custom shapes or morphologies in a user-defined region of the reciprocal space for all possible grazing incidence angles and sample rotations. This flexibility allows a straightforward study of a wide variety of possible polymer topologies and assemblies whether embedded in a thin film or a multilayered structure. Hence, this code enables guided investigations of the morphological and dynamical properties of relevance in various applications. The current parallel code is capable of computing GISAXS images for highly complex structures and with high resolutions and attaining speedups of 200x on a single-node GPU compared to the sequential code. Moreover, the multi-GPU (CPU) code achieved additional 900x (4000x) speedup on 930 GPU (6000 CPU) nodes.

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Dewetting of Confined Diblock Copolymer Films Studied by SERGIS

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Confining a polymer blend will induce morphologies different from those in the bulk. A method to confine the geometry is investigating very small droplets containing a polymer mixture. This can be accomplished by annealing a spin-coated thin film resulting in dewetting. The islands that are created will have a diameter and inter-island distances much larger than their height. Samples consisting of a blend of deuterated polystyrene (dPS) and polyparamethylstyrene (PpMS) were studied by AFM, neutron reflectometry and GISANS [1] and compared with the results of a destabilized sample consisting of a dPS homopolymer. For both samples the GISANS data showed a peak corresponding with the distance between the droplets. For the blend sample an extra peak at larger wave-vector transfer appeared that was interpreted to correspond to the length scale of the internal structure.

In the relatively new technique Spin-Echo Resolved Grazing Incidence Scattering (SERGIS) ultra-small angle scattering (as a result of in-plane structures perpendicular to the neutron beam) is coded by spin-echo. Here correlation ranging from tens of nanometres up to ten of micrometres are probed. The challenge of this technique is the fact that the spin-echo signal (variation of measured polarization due to the USANS) is generally much smaller than the specular signal (with polarization unity). Vorobiev *et al*. [2] studied similar dewetted polymer systems by means of SERGIS using the monochromatic instrument EVA at ILL, and interpreted these data to be consistent with the GISANS results. We did several SERGIS experiments on the time-of-flight instrument OffSpec at ISIS and were up to now not able to reproduce the results of Vorobiev *et al*. In this contribution we will compare the monochromatic and time-flight method and discuss the apparent discrepancies. Furthermore, possible methods will be discussed that may overcome the intrinsic low signal-to-background problem in the SERGIS technique.

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Observing thin film process technology in-situ – the case of sputter deposition

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Thin-film-based devices play a very important role in advanced scientific and technological applications [1,2]. To name just a few are magnetic materials [3], sensors [4] and organic photovoltaic cells [5]. Typically several process steps are used to install such devices. They range from fluidic application to install polymeric layers [5] to vacuum deposition methods [6] to coat metallic and oxide layers [1]. It is therefore of utmost importance to understand these individual process steps leading to the full device in order to be able to tailor them on the nanoscale [5].
Sputter deposition is one of the most widely used deposition methods to create metallic coatings. With grazing incidence small-angle x-ray scattering (GISAXS) being a very powerful method to investigate processes on the nanoscale and *in-situ*, we therefore combined *in-situ* sputter deposition and GISAXS at the MiNaXS/P03 beamline (PETRA III, DESY) [7]. We hereby follow a two-fold approach. On the one hand, we focus on deposition rates approaching industrial-style rates [8]. Here, we are able to follow with high time resolution the build-up of the metal layer and identify different growth rates. Especially we are able to deduce such important parameters as contact angles of the nanoclusters with the underlying surface. On the other hand, the nucleation process is still not easily observed. However, the initial nucleation behaviour strongly influences the metal layer nanostructure. We therefore present a first approach using GISAXS to observe these very early stages of sputter deposition [9].

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Multi-lamellar and non lamellar lipid-aquous structures at interfaces.

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Biological membranes do not only occur as bilayer structures, but bilayers have also been shown to, depending on the lipid composition, curve into intriguing 3D structures. It is clear that such lipid systems have come to use in food and pharmaceutical applications. Understanding the biological implication as well as the application of such interfaces requires the development of well-defined model system. Creation of such lipid structures can be obtained by adsorption of large aggregates, by creation or breakdown of structure, by phase transition within the interfacial layer but also invoked by bulk phase transition. Some recent findings, where we used neutron reflectometry in combination with bulk studies such as small angle neutron and X-ray scattering to reveal the layer structure and deposition mechanism. The focus will be on discussing the means to control the deposition of lipid based liquid crystalline nano-particles and how e.g. the formulation and surface properties can be used for this purpose. The challenge is to probe such structures at interfaces.

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Analysis of X-ray photon correlation spectroscopy data acquired in grazing incidence small angle scattering geometry

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Scattering experiments based on coherent X-ray beams have opened new possibilities for the investigation of soft and hard condensed matter [1]. Combining coherent illumination in grazing incidence and using the X-ray photon correlation spectroscopy (XPCS) technique [2], information about the dynamics of a surface can be obtained. We have recently used this approach to study the ageing dynamics of a self-organised surface [2]. The dynamical information is obtained from the temporal correlation of the intensity fluctuations. The intensity correlation function is related to the dynamic structure factor of the surface. In this talk, the experimental requirements and the details of the GI-XPCS data analysis will be presented, starting from raw 2D detector images up to the calculation and analysis of the correlation functions. Special attention will be paid to the issues (drifts, normalization issues, etc.) that may affect the analysis and produce wrong results.

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Directly programmable data analysis kit (DPDAK) for online analysis of high throughput 2D scattering data

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DPDAK is an open source (GPL) general purpose software framework for preliminary online analysis of large data sets resulting from high throughput scattering experiments using large area detectors at synchrotron radiation sources. The framework is independent of the beamline and/or experiment control software in order to be exchanged between different centres. Therefore, the software package is developed in Python programming language and avoids commercial and license-based libraries. An easy plugin interface allows extending the software with new procedures for reading, processing, displaying and exporting data. It is available at https://dpdak.desy.de.

DPDAK supports a wide range of detector formats (e.g. TIF, CBF, MCCD, EDF) and comes with evaluation methods for SAXS and GISAXS experiments like integration methods, peak positions, peak widths, intensity moments, and/or power laws [1,2]. It includes displays for detector images, plots and 2D maps. Data can be exported as spread sheet text files or saved in an HDF5 file including all processing parameter.

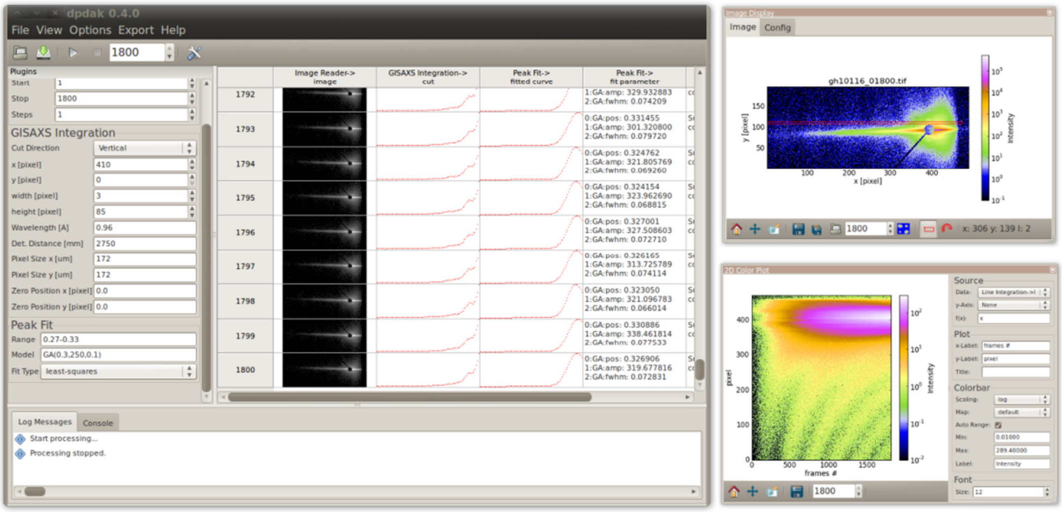

Figure 1 - The main frame (left) showing a typical analysis session with 1800 processed GISAXS detector images. Additional displays (right) are giving a detailed view on the data (e.g. detector image, 2D color map of cuts).

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Upgrade to the I22 Scattering Beamline to perform Grazing GI-SAXS experiments

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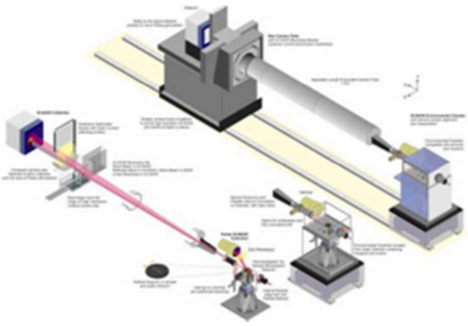
I22 is the principal beamline for small angle X-ray scattering at Diamond, with a flexible layout to conduct a wide range of transmission studies. To expand the capabilities of the beamline to thin films and surfaces, a major upgrade programme is currently underway to give high quality, versatile and dynamic grazing-incidence SAXS capabilities [1, 2]. This is ideal for studying inorganic and soft matter systems with thicknesses ranging between hundreds and tens of nanometres, such as organic photovoltaic (OPV) polymer blends [3, 4], block co-polymer systems, ceramic surfaces or aligned nano-fibres.

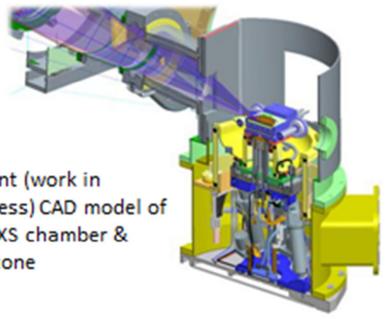
However, working at grazing incidence requires far more involved and accurate sample manipulation than for transmission SAXS. Facilitating dynamic studies of soft matter systems complicates this manipulation further, and also necessitates a complex environmental housing. The upgrade programme consists of two parts. Firstly, a dedicated GI-SAXS environment chamber that will be equipped to perform a range of high-resolution *in-situ* studies of a wide range of thin-film systems. With internal sample alignment, the chamber may be evacuated or helium filled, will be solvent vapour and moisture compatible, and aims to allow:

• The automated imaging of a rack of *ex-situ* prepared samples.
• The *in-situ* casting of a thin film (dip casting and doctor blading).
• The *in-situ* thermal / solvent annealing of a thin film.
• The collection of partial 2D GI-WAXS data, beyond the GI-SAXS nosecone.

Secondly, the detector table and beamstop module is being replaced with a new module containing several beamstops with independent internal motion.

Original proposal illustration showing both chamber and beamstop concept





Current (work in progress) CAD model of GI-SAXS chamber & nosecone

The design phase is now underway. Installation and commissioning of the first phase (beamstops and lower chamber with hexapod) will hopefully occur in the August-September 2013 shutdown. Detailed design of the second phase (the sample environment chamber and sample mount / manipulation modules) will commence in the summer.

This talk will present an overview of the upgrade as it currently stands, and highlight the upcoming research opportunities it presents.

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Abstracts: Poster Presentations

Spin echo grazing incidence scattering (SERGIS) of buried structures

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The relatively new technique of SERGIS has proved powerful in probing periodic grating samples and there is now some level of maturity in modelling this data.[1] So that it is now possible to extract meaningful lengthscale and periodicities from these real space neutron measurements. These recent experiments wanted to assess to what extent the SERGIS signal from a buried periodic structure could be measured. We used glassy thin film deuterated PMMA polymer samples floated onto a grating structure to make a buried periodic structure. We will present SERGIS data for two thicknesses of buried gratings as well as a dPMMA film that has been heated above T_g and so conformed to the buried grating structure and so gave an added periodicity to the SERGIS signal.

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Characterisation of Buried Conjugated Polymer Interfaces by Off-Specular Neutron Reflectivity

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Characterisation of polymer-polymer interfaces, where one or both of the polymers is conducting or semiconducting (conjugated) is an interesting and technologically important subject. Such interfaces are at the heart of plastic electronic devices, as they are the site of charge separation/recombination in photovoltaics/light-emitting diodes (LEDs), and are the location of the charge conduction channel in field effect transistors (FETs). Interfaces that contain these relatively stiff, liquid-crystalline polymers can be significantly different to the more familiar amorphous polymer-polymer interfaces. For example, very broad interfaces in conjugated-polymer/conjugated-polymer systems (heterojunctions) of direct relevance to LEDs and photovoltaics have recently been discovered [1]. Neutron reflectivity offers the opportunity for non-destructive characterisation of these buried polymer interfaces. To date, we have used specular neutron reflectivity to characterise the structure of conjugated polymer interfaces found in LEDs [1] and FETs [2]. However, detecting specularly reflected neutrons only allows us to measure the root-mean-square (rms) roughness of the interface averaged over a macroscopic lateral distance (the in-plane coherence length of the neutrons). There are two contributions to the rms roughness of a polymer-polymer interface; i) the so-called 'intrinsic interfacial width' due to mixing of the polymers at a molecular level and ii) lateral roughness due to deviations of the interface position from the plane of the substrate (eg due to capillary waves at a liquid-liquid interface). Here we present recent work in which we have collected both specular and off-specular neutrons, allowing us to probe structure in the plane of the interface and to distinguish intrinsic interfacial broadening from longer wavelength lateral roughness. We model the off-specular scattering using the Distorted Wave Born Approximation (DWBA) [3], applied to a bilayer geometry.

In this work we investigate a model conjugated polymer/amorphous polymer interface (poly(9,9-dioctylfluorene (F8) on deuterated poly(methyl methacrylate) (PMMA)). We examine the structure of this interface by systematically varying the film thickness, which strongly impacts on the amplitude of the lateral interface roughness, and allows a more complete analysis of the relative contributions of intrinsic mixing and lateral roughness. Our findings are compared with theoretical predictions, using self-consistent-field-theory, for flexible and semiflexible polymer-polymer interfaces.

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Grazing incidence small-angle scattering at ESS

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The ESS will be the world's leading long pulse neutron source, with 7 out of the first instrument suite of 22 expected to come operational in 2019. During the Pre-construction phase 2010-2012, the Instrument Design Update project has explored a number of reflectometer and SANS instrument concepts capable of Grazing Incidence small angle scattering studies of surface structures. These instruments and their applications will all benefit from the high flux and flexibility offered by the ESS long pulse structure and should realise at least an order of magnitude performance gain over existing instruments.

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Depth-sensitive time-of-flight small-angle neutron scattering

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Surface effects in liquids and polymers gain enormous in importance with decreasing size of mechanical components and biological applications. A detailed knowledge of the solid-liquid boundary condition leads to the development of smart coatings, e.g. self-cleaning surfaces, and is an important ingredient for the understanding of the folding of proteins in the vicinity of cell membranes.

However, on the molecular scale the solid-liquid boundary is difficult to probe directly and non-destructively. As an example, AFM, SFA or optical techniques need direct contact to the surface or tracer particles, respectively. On the other hand it has been shown that scattering techniques are an excellent possibility to contribute in this context, since they are non-invasive and non-destructive, with minimum perturbation of the sample. In particular, neutrons offer a high penetration power for most engineering materials combined with a large scattering power of light elements, like e.g. hydrogen and deuterium. This makes them ideal candidates for probing solid-liquid interfaces in a beam geometry where the neutron beam passes through a silicon wafer and gets reflected from the solid-liquid boundary.

The goal of this work is to extract the near-surface structure of polymer micelles close to interfaces with different surface energies. We have used Pluronic F127 ((C2H4O)99-(C3H6O)65-(C2H4O)99), dissolved to 20% in deuterated water. The polymer micelles forming in the solution have been investigated with the instrument Sans2D at ISIS (UK). A wavelength spread was used (wavelength spectrum: approx. 1 - 18Å) at a fixed angle of incidence allowing a variable penetration depth between 6.5nm and 20µm of the neutrons into the liquid.

The refractive index of the sample and the penetration depth of the neutrons are calculated via the coherent scattering lengths, the absorption and the incoherent scattering cross sections of the elements in the sample.

We report on the depth resolved structure of the polymer micelles, which shows a pronounced dependency on the surface energy of the solid boundary.

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Grazing incidence techniques on XMaS

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The universities of Liverpool and Warwick operate the EPSRC funded XMaS beamline located at the ESRF in Grenoble, France. The project has been delivering a broad, inter-disciplinary research programme to UK and EU users since 1997. Its research programme covers both fundamental blue skies and applied materials research in emerging technologies. It tackles a broad range of scientific challenges including soft matter, surface electrochemistry, modern magnetic materials, conservation of historical artefacts and even medical materials. Although the beamline was originally designed for the exploration of magnetic materials using scattering techniques, surface science studies represent a third of the beam time activities.

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GISAXS simulations for laboratory instrumentation

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New detector technologies, together with high resolution optics and precision goniometers, are enabling GISAXS experiments to be performed on laboratory instruments. These early experiments are not without their challenges. We have successfully collected 2D GISAXS data for a number of inorganic thin films variously containing nano-particles, nano-pores and nano-layers. On a laboratory instrument we are potentially dealing with low signal, low dynamic range, and image distortion for flat detectors at small radii.

In this poster we consider the implications of these experimental challenges for comparison of 2D data with simulated images. We are working with a prototype software which simulates a 2D non-coplanar (Qy,Qz) reciprocal space map, for single wavelength radiation, incorporating Distorted-Wave Born Approximation (DWBA) or semi-kinematical diffuse scatter algorithms (1).

We consider the geometrical and resolution limitations of the experiment, together with measured dynamic range and appraise the outcome for simulation of our experiment.

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Inert nano-reactors or dynamic micelle interfaces? $CaCO_3$ precipitation from microemulsions

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Reverse microemulsions are, thermodynamically stable suspensions of water droplets in oil i.e. micelles that are stabilised by an interface surfactant. Water droplets are typically 1-50 nm in diameters and can carry dissolved salt ions and exchange their content upon collisions, which lead to mineral precipitation. These droplets are believed to act as "nano-reactors" because precipitation occurs in the water pools shielded by the surfactants from the oil phase.

Here, we show how we can use microemulsions to elucidate the formation of $CaCO_3$ phases and stabilise initial amorphous stages. Micelles are used to confine volume in which nucleation and growth occurs. Mixing of two distinct microemulsions containing Ca^{2+} and CO_3^{2-} ions leads to a reproducible method to make nano- and micro-sized, monodisperse particles. However, there is no correlation between the initial droplet size and the size of solid $CaCO_3$ particles, which can be considerably larger than the original micelles. Therefore, the notion of a "nano-reactor" may in this case be inaccurate, because it implies the formation of an inert, impenetrable water-surfactant-oil interface that limits the growth to a single droplet.

By using time-resolved and *in situ* small X-ray scattering and high-resolution imaging we demonstrated that crystalline $CaCO_3$ grows through a slow but progressive agglomeration of the amorphous particles of the size of original micelles. Hence, the process is not limited only to precipitation inside of individual water droplets. Upon destabilisation of the original salt-ion carrying micelles, new water-crystal-surfactant interfaces are created. These constitute a nucleus and they direct further growth. The formation of this interface is crucial in stabilising amorphous $CaCO_3$ in the form of nanoparticles (10-100 nm) and this also slows down or prevents further growth and crystallization above ca. 200 nm.

We believe that these findings are relevant for understanding of the $CaCO_3$ growth mechanisms occurring at water-nonpolar liquid interfaces in natural and industrial environments (e.g. preventing scale formation). Microemulsions could also be a good analogue model system for mineralization of coccolith plates formed within the cell vesicles produced in the Golgi apparatuses of coccolithophores.

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HipGISAXS: A High Performance Computing Code for Simulating Grazing Incidence X-Ray Scattering Data

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Grazing Incidence Small-Angle Scattering (GISAXS) is a valuable experimental technique in probing nanostructures of relevance to polymer science. New high-performance computing algorithms, codes, and software tools have been implemented to analyze GISAXS images generated at synchrotron light sources. We have developed flexible massively parallel GISAXS simulation software ``HipGISAXS'' based on the Distorted Wave Born Approximation (DWBA). The software computes the diffraction pattern for any given superposition of custom shapes or morphologies in a user-defined region of the reciprocal space for all possible grazing incidence angles and sample rotations. This flexibility allows a straightforward study of a wide variety of possible polymer topologies and assemblies whether embedded in a thin film or a multilayered structure. Hence, this code enables guided investigations of the morphological and dynamical properties of relevance in various applications. The current parallel code is capable of computing GISAXS images for highly complex structures and with high resolutions and attaining speedups of 200x on a single-node GPU compared to the sequential code. Moreover, the multi-GPU (CPU) code achieved additional 900x (4000x) speedup on 930 GPU (6000 CPU) nodes.

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In situ Observation of the Gold Cluster Growth on the Block Co-polymer Substrates Using Sputter Deposition and μGISAXS

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Hybrids consisting of block co-polymer and nanoparticles are one of the most favourable nanocomposites.[1] By tailoring the structures, the nanocomposites can offer us enhanced optical, electronic and magnetic properties, and they are promising to prepare devices, such as organic light-emitting diodes, solar cells and magnetic storage medias.[2-4] However, the successful wide application of these advanced materials in the industrial and domestic fields meets some obstacles, for example, the productive efficiency. Hence the *in situ* study of the structure evolution and exploring the inherent fast assembly of nanoparticles are essential because it can help us improve the productive efficiency without sacrificing performance.

With the aid of a highly automated sputter equipment and high photon flux and fast time-resolved data acquisition in MiNaXS/P03 beamline (PETRA III, DESY),[5, 6] we combined the sputter deposition with microbeam grazing-incidence small angle X-ray scattering (μGISAXS) and *in situ* studied the gold cluster growth on the polystyrene-*block*-poly(epsilon-caprolactone) (PS-*b*-PCL) thin films. The choice of the PS-*b*-PCL diblock co-polymer thin films was due to its simple microphase structure and rich topography structures under heat treatment.[7] Meanwhile, μGISAXS can offer us direct information about the structure evolution on the nanoscale.[8] By controlling the substrate temperature, we studied the assembly process of the gold nanoparticles on PS-*b*-PCL diblock co-polymer thin films with different topographies. The results focusing on the influence of the substrate topography on the clustering of gold and the efficiency of the gold-assembly at different temperatures are presented.

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DOMAIN CORRELATIONS IN COMPLEX STRATIFIED MAGNETIC STRUCTURES – SIMULATION/FITTING OFF-SPECULAR SCATTERING

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Due to their scientific and technological relevance, artificial magnetic heterostructures have attracted much interest in material science. Polarized specular neutron reflectometry and off-specular neutron scattering at grazing incidence has proved to be decisive in the identification of magnetization in various spintronic materials such as interlayer-coupled or exchange bias systems. The magnetic domain structure in a stratified system is dependent on a number of parameters, such as the interlayer coupling, the magnetic anisotropy, interface roughness and the external magnetic field and exhibits hysteretic behavior.

Off-specular neutron reflectometry is used to characterize thin films both in the out-of-plane and in-plane directions, namely to determine the statistical properties of the interfaces, and the magnetic domain structure. Its use is at least as much limited by the lack of widely available, user friendly, and open-source modelling software as by the available neutron flux at the current neutron sources.

Currently, the best interpretation of the off-specular scattering can be accomplished in the distorted wave Born approximation. In DWBA the scattered intensity is expressed in terms of the average layer structure and the statistical correlation functions specifying the lateral inhomogeneities (nuclear and magnetic). These parameters of the magnetic structure are comprised in the inter- and intra-layer domain correlation functions.

Symmetry properties of the intra- and interlayer domain correlation functions were exploited in order to minimize the number of relevant correlation lengths in the 2D fitting of the diffuse scatter.
The capabilities of the FitSuite code [1] will be presented in modelling domain structures in stratified systems and in simulating and fitting 2D offspecular intensity maps. The magnetic domain structure will be investigated using FitSuite simulations of off-specular neutron scattering experiments on two strongly antiferromagnetically coupled [Fe/Cr] epitaxial superlattices: a) in a structurally homogeneous periodic multilayer and b) a similar multilayer with the surface Fe layer lithographically structured into stripes [2].

In the unstructured ML sample (a), ^{57}Fe(2.6nm)/Cr(1.3nm)/MgO(001) following in-plane saturation, patch domains form with AF order through the ML stack [3]. Upon unsaturation a spontaneous irreversible increase of the average domain size occurs, ("domain ripening"), which takes place in a narrow range of the external magnetic field strength. These phenomena are quantified by the in-plane correlation length of the layer magnetization extracted from the off-specular spectra by fitting the 2D intensity map to a two domain model.

In the laterally structured sample (b), the domain structure is dependent on whether the external magnetic field is applied along or perpendicular to the Fe-stripes. Due to lateral structuring, the diffuse scattering is considerably enhanced especially near the antiferromagnetic reflections, indicating maintained antiferromagnetic correlations and columnar domains. Interpretation of the data requires at least five different types of domains.

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