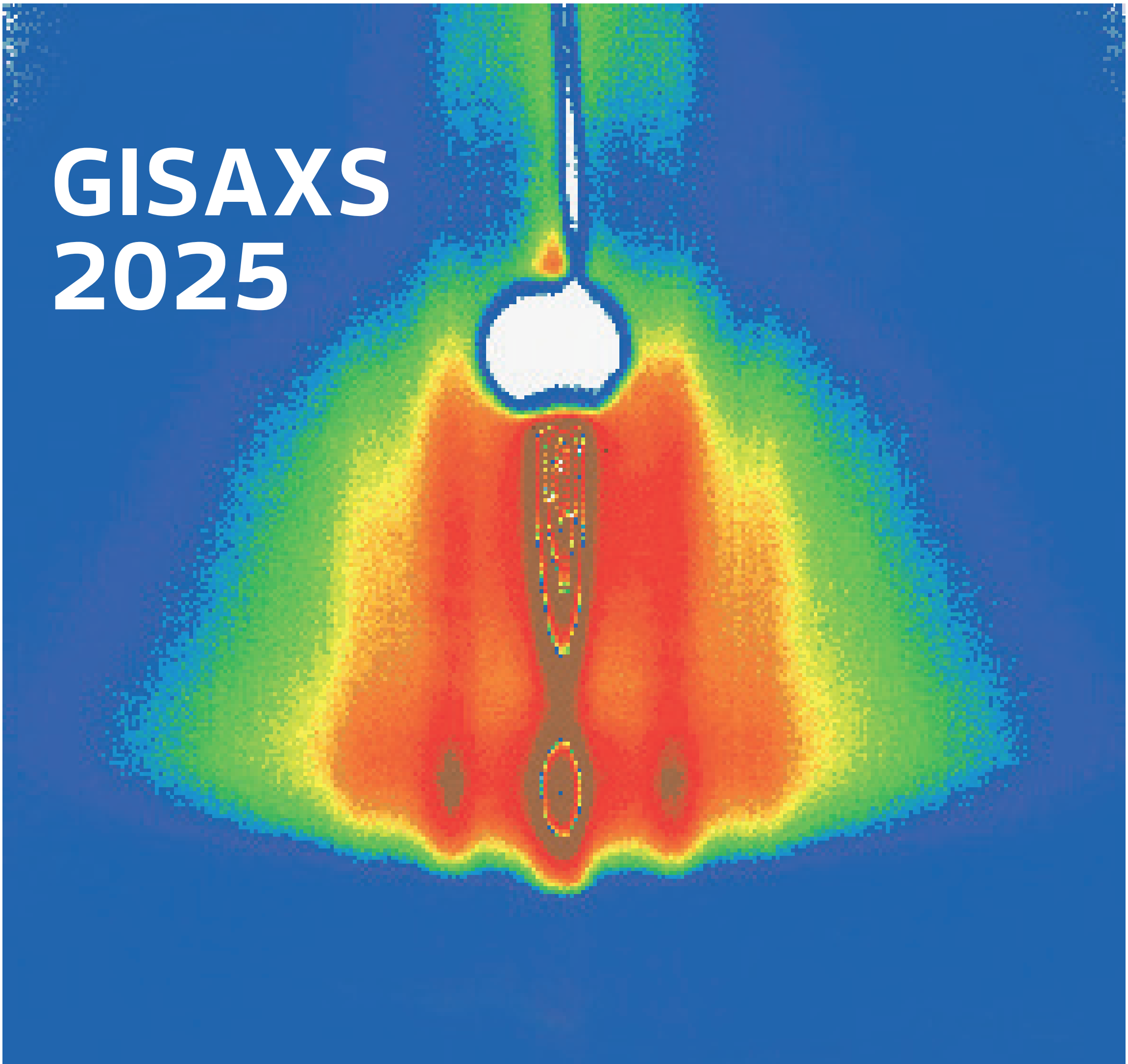
GISAXS 2025



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Understanding the crystalline and amorphous morphology of organic solar cells using grazing-incidence small-angle X-ray and neutron scattering techniques

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Organic photovoltaic molecules or polymers typically form semicrystalline thin films. It is widely recognized that the bulk heterojunction (BHJ) morphology of organic photovoltaic thin films, which consists of both crystalline and amorphous regions, plays a crucial role in determining the performance of devices. However, understanding the intricate three-dimensional multi-length scale morphology of these thin films remains a grand challenge. In this talk, we will present our recent progress on decoding the complex BHJ morphology of OPVs and developing strategies to control the morphology to enhance device performance. In addition to employing conventional techniques like GISAXS/GIWAXS, we will introduce two innovative methods: GTSAXS, which allows for the quantification of vertical nanomorphology, and GISANS combined with deuteration, a technique used to detect amorphous phase structures. Armed with these state-of-the-art scattering techniques, we investigated the optimal active layer morphology for OPVs, aiming at understanding the impacts of amorphous and crystalline phase structures on device performance and to advance the practical applications of these devices. Furthermore, these scattering techniques can also be applied in material science, chemistry, biology and condensed matter physics studies. By modifying the wavelength of the probing beam and the experimental geometry, a variety of sample types, such as solutions, powders, surfaces and thin films, can be studied, covering wide length scales as well as versatile dynamic and kinetic behaviors.

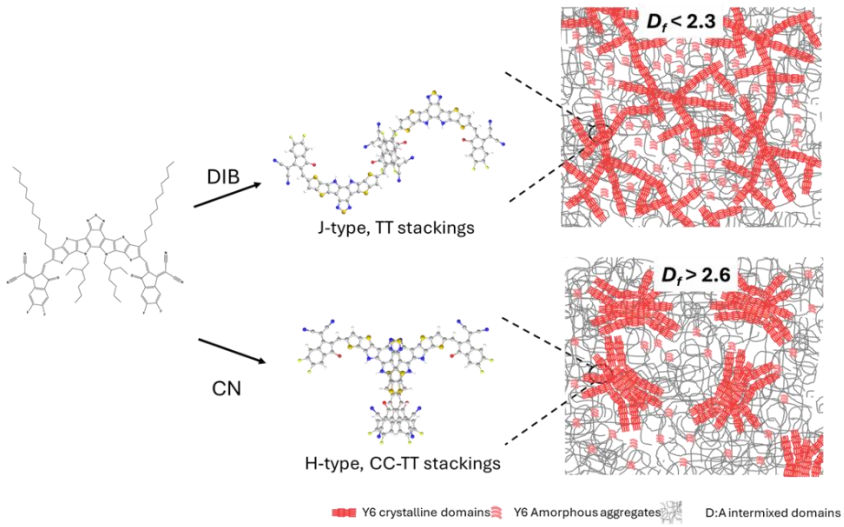

Figure 1: Multi-length-scale packing structures of Y6 with different additives [1].

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A Basic Introduction to GISAXS and GISANS

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The investigation of nanostructures at surfaces, interfaces, and in thin films requires dedicated analytical techniques, which provide information from a molecular to a mesoscopic scale. [1] Grazing incidence small-angle x-ray and neutron scattering (GISAXS and GISANS) overcome the limitations of conventional small-angle x-ray and neutron scattering with respect to extremely small sample volumes in the thin film geometry by the use of a reflection geometry [2-4]. GISAXS/GISANS involve a combination of two techniques, GID (grazing incidence diffraction), which uses a reflection geometry to obtain surface and near-surface sensitive scattering, and SAS (small-angle scattering), which measures structures of 1 - 100 nm length in normal transmission mode. They are non-destructive structural probes and do not require special sample preparation. GISAXS/GISANS yield excellent sampling statistics (averages over macroscopic regions to provide information on nanometer scale) and provide information on scattering object geometries, size distributions, and spatial correlations. However, in GISAXS/GISANS experiments, the high demand for collimation requires the use of high-flux X-ray and neutron sources.
After a basic introduction to the GISAXS/GISANS techniques, several different examples of thin nanostructured polymer films are presented to illustrate the possibilities and challenges of GISAXS/GISANS. In addition, the challenges and potentials of in-situ studies during printing and in operando studies of organic and perovskite solar cells are presented and discussed in detail. [5-7]

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GISAXS of Obliquely Deposited Co_2FeAl Thin Films on Patterned Substrates

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The artificial tailoring of magnetic anisotropy by manipulating surface and interface morphology is attracting widespread interest from the application point of view in spintronic and magnetic memory devices. We present oblique angle deposition on a nano-patterned rippled substrate as a geometrical route of inducing in-plane uniaxial magnetic anisotropy (UMA) in magnetic amorphous films. For this purpose, CFA films and ripple Si substrates have been taken as a model system to demonstrate the same. Here, nano-patterned substrates are prepared by low-energy ion beam sputtering (IBS), above which films are deposited obliquely normal to the ripple directions. Grazing incidence small-angle (GISAXS) and wide X-ray scattering (GIWAXS) measurements were conducted at the P03 beamline, PETRA-III, DESY, for morphological and structural investigation. GISAXS measurements of the rippled films exhibited periodically aligned vertical streaks on either side of the specular plane (Fig.1), indicative of lateral periodicity. The asymmetric distribution of scattered intensity and the presence of inclined fringes reflect the formation of inclined columnar structures [1,2]. A clear anisotropy in the growth behavior has been observed due to the inhomogeneous in-plane organization of adatoms in the form of columns. The increased shadowing effect in the films deposited obliquely normal to the direction of the ripple patterns causes preferential coalescence of the columns along the substrate ripples, resulting in strong in-plane magnetic anisotropy in the film. This peculiarity in magnetic behavior is addressed by considering the morphological anisotropy governed by enhanced shadowing effect, the shape anisotropy, and the dipolar interactions among the magnetostatically coupled nanocolumnar structure. Our results demonstrate that deposition geometry and initial surface topography have a direct and crucial bearing on the interfacial microstructure, which can be utilized for controlled tuning of UMA by preselecting a suitable configuration.

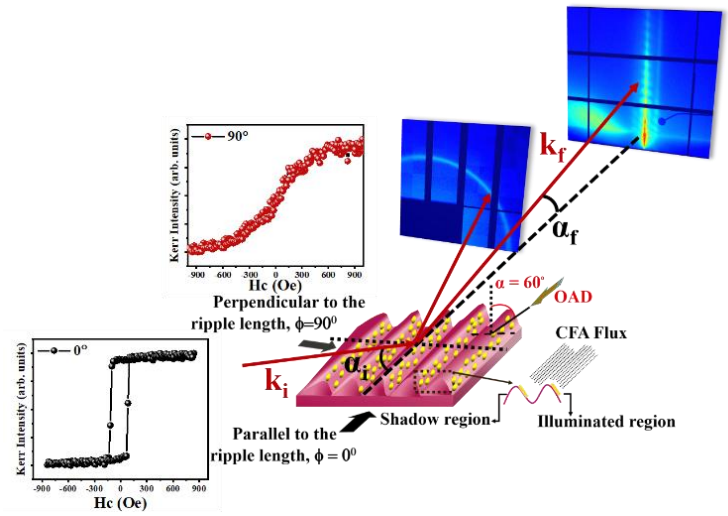

Figure 1: Schematic representation of GISAXS and GIWAXS geometry, along with MOKE loops of 50 nm CFA film deposited on rippled Si Substrates

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Neutron Scattering from Soft Matter at Interfaces – Challenges and Potential

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Grazing incidence scattering of neutrons is useful for exploring structure at interfaces of bulk phases of materials and there are key differences, both advantages and disadvantages, to other available techniques such as microscopy and X-ray methods. These will be illustrated with examples of particulate colloidal dispersions, concentrated surfactant phases and lipid adsorption to structured surfaces. An example in Figure 1 showing scattering from 9 wt.% polystyrene latex particles (radius 72 nm) dispersed in D_2O next to the oxide layer on a silicon crystal illustrates several important features [1]. The broad distribution of wavelengths necessary for adequate intensity significantly influences the penetration depth of the incident beam such that signal only from an evanescent wave is not readily observed but rather is dominated by scattering from the incident beam penetrating the liquid sample in the near surface region to depths of 100's of nm. The mean depth that is probed can be calculated readily from the total cross-section of the materials that for neutrons, in contrast to X-rays, is usually dominated by scattering rather than absorption.

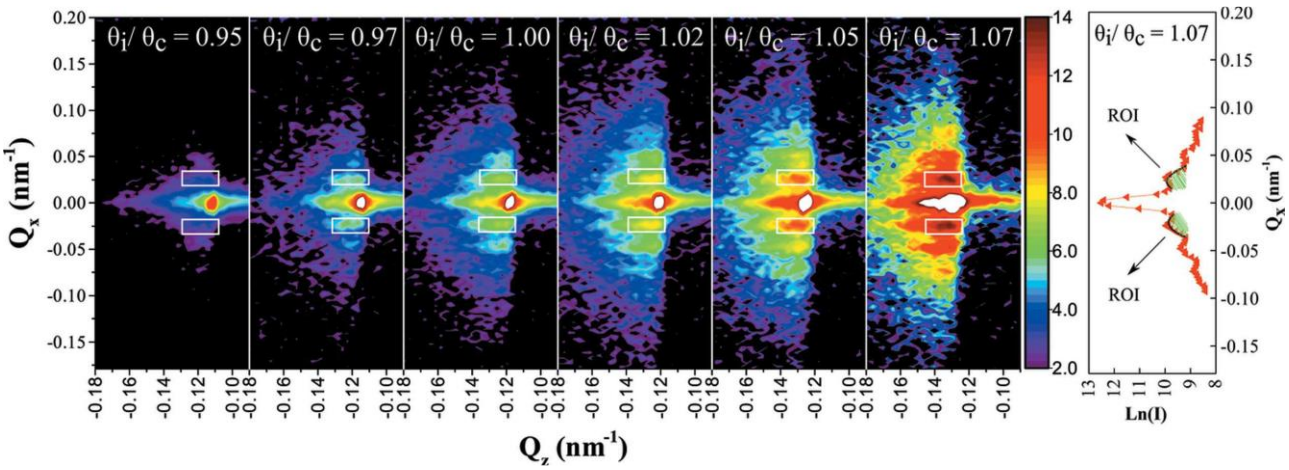
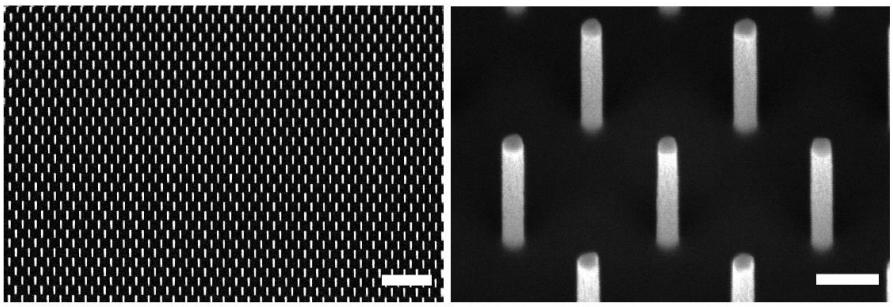


Figure 1: Scattering intensities recorded close to the critical angle from 9 wt.% PS3 latex in D_2O [1] against a silicon surface. The critical angle for $\lambda = 1.4$ nm was 0.8°. The intensities are normalized to the measurement time and shown on a $\log_e$ scale. The vertical strip along the central part of $\mathbf{Q_x}$ on the right side of the figure shows the integrated peaks as an example for $\theta_i / \theta_c = 1.07$.

The information that is obtained from such experiments usefully identifies that structure in the few micrometres near the solid surface is modified from that in the bulk of the latex sample. Other similar experiments have been used to study the pre-wetting of a lamellar phase of an anionic surfactant at a solid surface that is in equilibrium with a lower concentration in the bulk solution [2].

As many interfaces are not naturally flat or smooth, it is of interest to study a variety of structures at surfaces and how they are modified by surfactants, lipids, polymers and biopolymers. The induced orientation of surfactant multilayers and how they are influenced by temperature and surface roughness have been studied using measurements of rocking curves for a concentrated non-ionic surfactant in D_2O [3]. One development has been to use lithographic methods to create a highly ordered array of nanowires on a silicon substrate [4]. This is shown in Figure 2 and has been used for experiments probing lipid and protein adsorption to the highly curved surfaces of the pillars. Experiments on such highly ordered arrays have illustrated several interesting features of grazing incidence scattering.



(a) (b)

Figure 2: Nanowires imaged with SEM at a 30° tilt of the stage. The scale bar in (a) is 2 µm and in (b) 200 nm. The picture shows only part of the 65 mm × 65 mm substrate [4].

The scattering from the nanowires can be made to 'disappear' by placing the substrate in contact with a mixture of H_2O and D_2O with a scattering length density of 2.07×10^{-6} Å^{-2}. Grazing incidence neutron scattering results are illustrated as raw data in Figure 3 where the images of the scattering from bare nanowires and after exposure to lipids (20:80 wt.% DOPE:DOPC) as a vesicular dispersion. The spots on the left-hand side of the direct beam are clearly visible when there is adsorption to the surface.

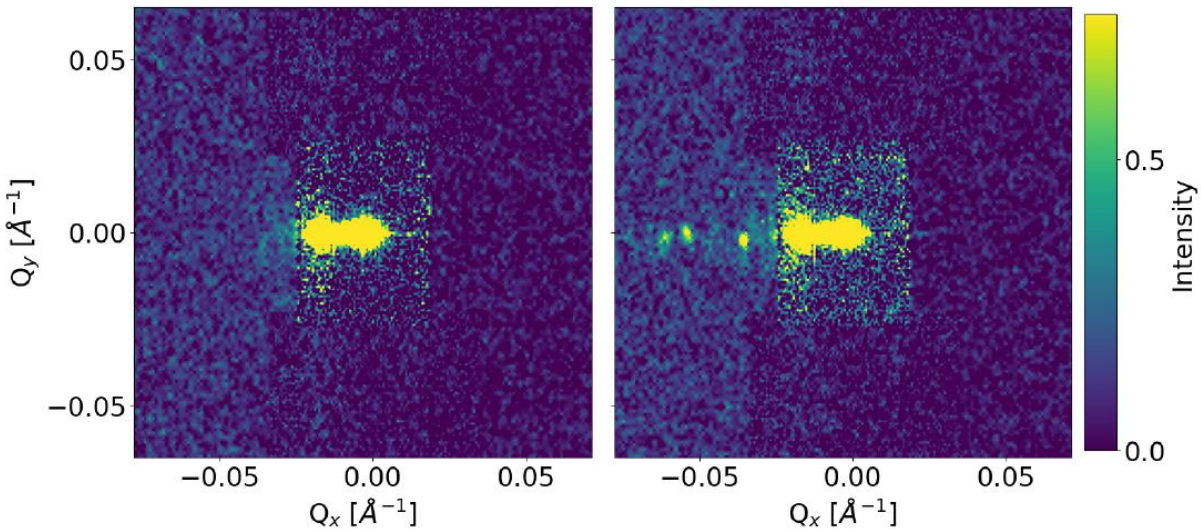


Figure 3: Scattering from bare nanowires (left) and with a lipid layer (right) measured in water matched to silicon. Measurements at NCNR with 6 Å wavelength and 0.55° incident angle. With just the thin oxide layer at the surface, scattering is insignificant but with lipid adsorption the nanowires are clearly visible [4].

There are several challenges in the quantitative interpretation of these results that can be compared with measurements in different contrasts, measurements of the substrate with X-rays and complementary techniques such as reflection measurements. The pattern of intensity variation with $\mathbf{Q_x}$ is dominated by the form factor of the nanowires as the diffraction peaks from the lattice are too finely spaced to be resolved. However, the pattern is reinforced by the structure factor that has maxima that are closely spaced in preferred directions. Complementary GISAXS studies illustrate the issues of angular resolution and alignment.

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Combining Electrochemistry with In Situ GISWAXS

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Advanced in situ characterization techniques, such as grazing-incidence small- and wide-angle X-ray scattering (GISWAXS), are essential for gaining deeper insights into electrochemically driven nanoscale processes occurring during material synthesis and operation. This work presents recent progress in combining electrochemistry with in situ and in operando GISWAXS to investigate and optimize nanostructured materials in three areas: (i) energy applications (e.g., fuel cells), (ii) electrochemical dealloying, and (iii) templated electrodeposition. These studies provide feedback essential for the rational design of nanostructures to improve performance.

(i) Electrochemical energy systems such as proton-exchange membrane fuel cells (PEMFCs) rely on nanostructured catalysts and whose performance depends on structural dynamics at the nanoscale. Here, in PEMFC catalysts, structural evolution during operation is a critical factor for activity and stability. We studied Pt_xNi_{100-x} bimetallic nanoparticles as PEMFC catalysts deposited on carbon supports under accelerated stress tests, revealing a dynamic interplay of dealloying, Ostwald ripening, and particle coalescence. These processes drive changes of the catalyst dimension and its aggregation, directly influencing catalytic performance [1]. The insights highlight strategies to design more effective fuel cell materials with improved lifetime, activity and reduced costs.

(ii) Electrochemical dealloying is a widely applied route to nanoporous metals. While Au–Ag systems have been extensively studied, other alloys remain less explored. Using in situ GISAXS, KMC simulations, and STEM, we investigated CoPd and AgAu alloys, in which CoPd showed faster front propagation and slower coarsening compared to Au-based systems, attributed to its higher dealloying potential and stronger interatomic binding [2]. These findings validate the rate-equation model of dissolution and surface diffusion and demonstrate the general applicability of the mechanism to binary alloys, opening paths to tailored nanoporous metals with tuneable activity.

(iii) We further applied in operando GISAXS to track structural changes during templated electrodeposition of mesoporous Pt films from lyotropic liquid crystalline (LLC) phases with Brij®56 [3]. Initially, LLC hexagonal phase is aligned parallel to the electrode, but during deposition a rapid Pt nucleation burst disrupted this orientation. Over several minutes, the structure reorganized into vertically aligned mesopores with ~8.5 nm spacing. These results provide the first operando view of LLC-guided deposition, revealing the dynamic interplay between template and metal species. The resulting freestanding Pt films with vertical pores hold promise for electrocatalysis, energy harvesting, and nanofluidics. Further, latest results of the electrodeposition of mesoporous platinum nickel films will be presented using Pluronic P123 as structure directing agent, as proposed by [4].

By combining electrochemistry and GISWAXS, we gain online access to the nanoscale mechanisms governing catalyst degradation, dealloying, and templated deposition (see Fig. 1). These studies illustrate the power of GISWAXS to link structure and function in nanomaterials. At the end, the developments in the field of SAXS after the upgrade of ELETTRA to ELETTRA 2.0 will be presented, which further extend capabilities for in situ/in operando nanostructural studies in general, but also for in situ electrochemical GISWAXS investigations.

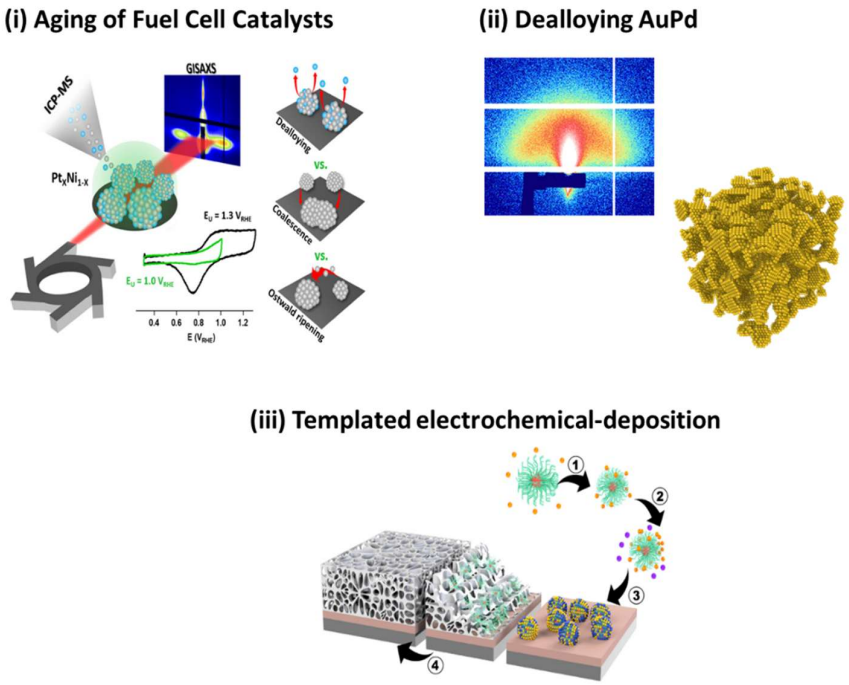

Figure 1: Schetch of the obtained results by in-situ electrochemical GISAXS investigations of (i) PEMFCs fuel cell catalysts, (ii) electrochemical dealloying of Co-Pd and Ag-Au systems, (iii) templated electrochemical-deposition

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Analyzing Large Grazing-Incidence X-Ray Scattering Datasets Using INSIGHT

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INSIGHT is a Python-based software package for processing and reduction of grazing-incidence wide- and small-angle X-ray scattering data (GIWAXS/GISAXS) [1]. It aims to be a solution to the emerging requirement for fast and flexible treatment of time-resolved GIWAXS/GISAXS data from synchrotron experiments. As a script-based and object-oriented package, it provides classes and methods to assist with all steps of data processing, from plotting of raw images to exporting publication-quality plots of the final results. Its main functionality is the transformation of raw two-dimensional detector images into reciprocal space, and the reduction to 1D cuts in horizontal, vertical, radial and azimuthal direction (see Figure 1). It focuses on efficient management of large datasets, aiming to simplify simultaneous processing, reduction and analysis of scattering data from in situ and operando studies on thin films. Full integration into the Python environment enables a wide range of user-defined workflows and combinations with other packages in the Python ecosystem. Special focus is placed on correcting offsets in intensity measured by the detector. Therefore, air attenuation, absorber attenuation and horizontal polarization of the X-ray beam is taken into account and corrected. Furthermore, errors in detector pixel positions caused by angular offset by the detector can be corrected, by allowing free rotation of the detector along three independent axes. However, there can also be dynamic factors that change the geometry over time when performing kinetic studies. Shifts in the sample-detector distance (SDD), for example due to sample translation or expansion during thermal treatment, can lead to apparent drifts in q-values of Bragg ring positions. If a stable calibrant such as indium tin oxide (ITO) is present, its peak positions can be used to reconstruct the true SDD and correct the geometric calculation, restoring the Bragg peaks to their correct positions. Furthermore, INSIGHT contains a module for indexing of crystallographic reflections and simulation of 2D GIWAXS patterns with multiple preferred orientations of crystallites (see Figure 2). This enables the disambiguation of complicated patterns of Bragg spots in 2D GIWAXS data and the determination of the true orientations of crystallites within the measured film. Together, these features make INSIGHT a comprehensive and versatile tool for the high-throughput analysis of complex GIXS datasets, facilitating deeper understanding of structural dynamics in thin-film materials.

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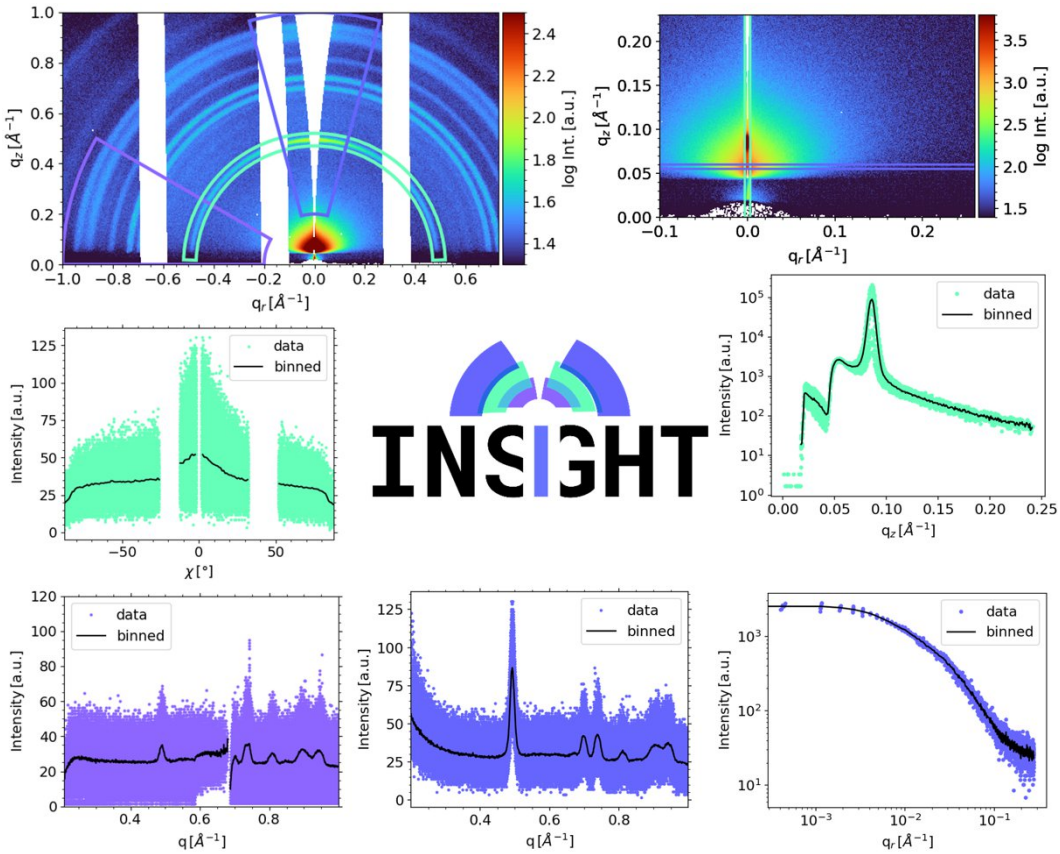

Figure 1: Demonstration of INSIGHT-based data reduction of GIWAXS (left) and GISAXS (right) detector data, showing the creation of azimuthal, radial, vertical and horizontal cuts.

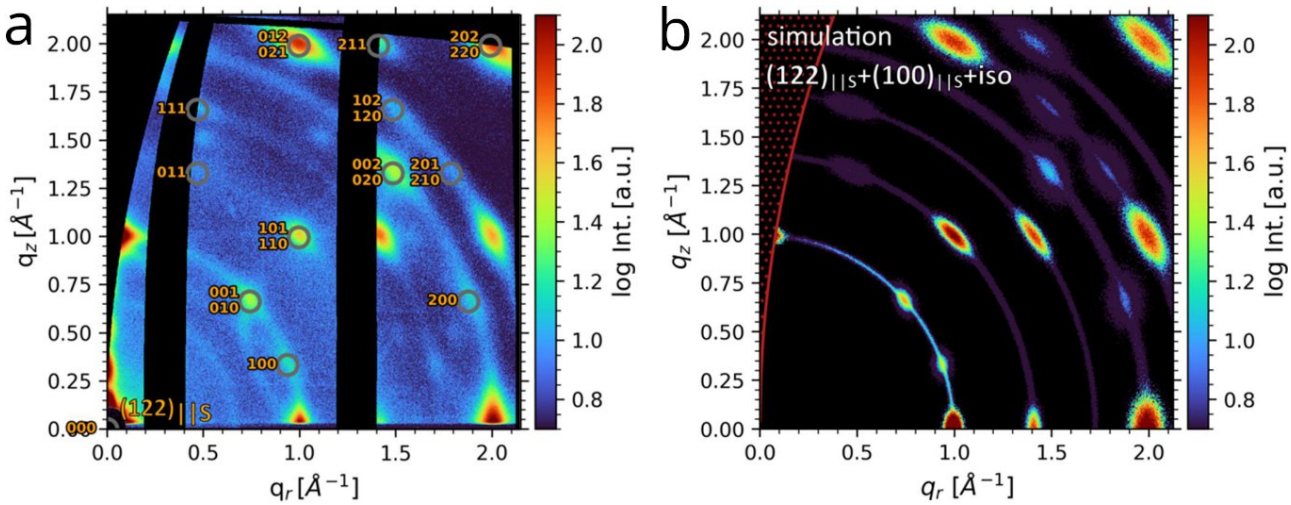

Figure 2: Demonstration of the INSIGHT-based GIWAXS indexing feature. **a**, Reshaped GIWAXS data with indices of Bragg spots overlayed. **b**, Simulation of GIWAXS image with predefined orientation matching that of **a**.

GISAXS and GISANS investigation on hybrid, bio-based thin films

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Flexible hybrid materials combining organic and inorganic, nanomaterials, such as polymeric colloids and metals, and bio-based templates are ideally suited for wearable electronics. Here, it is crucial to directly include scalable, large area fabrication technologies for biodegradable, eco-friendly, and, at the same time, high-performance materials. Such fabrication technologies should directly be based on green coating and printing approaches. Cellulose nanofibrils (CNF), extracted from wood, constitute a biomaterial par excellence to be used for green coating technologies. CNF can be suspended in water. In combination with their high-strength, they are ideal base materials for nanoporous templates. The void structure of these CNF networks ("nanopaper") facilitates the incorporation of filler particles (e.g. colloids, nanowires) and thus allows for their functionalization. To start with, spray-deposition – see Figure 1 – as scalable, highly efficient and rapid coating method for installing plasmon active silver colloid-CNF hybrid thin films for optoelectronic applications is presented [1].

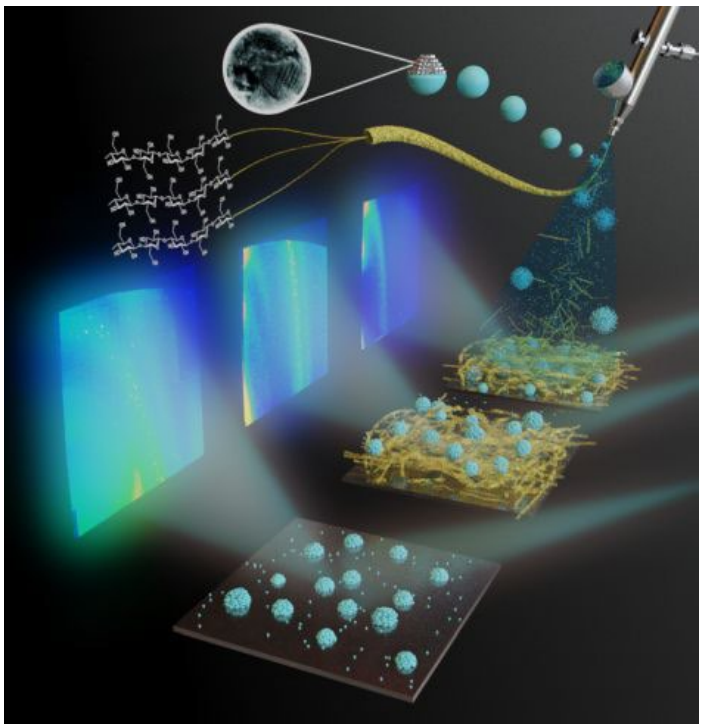

Figure 1: Preparation of cellulose nanofibril-colloid hybrid materials via spray deposition, with all preparation steps investigated by GISAXS.

The ink formulation as well as the choice of the layer-by-layer deposition greatly influences the homogeneity of the hybrid thin film. This is extended to conductive hybrid electrodes for flexible electronics [2]. Here, water-based CNF-silver nanowires ink formulations have been developed, and flexible nanocomposite electrodes via spray coating have successfully been developed. In a next step, by adding conductive polymer fillers, novel, micrometer-thin and freestanding hybrid nanocellulose foils have been used for 3D organic electronic applications, exploiting the high strength of CNFs and their water-processability [3]. Structural colors have been incorporated in nanopapers by adding core-shell polymer colloids into the nanoporous network. Scalable slot-die printing was employed to achieve dense CNF thin films, where the colloidal sub-phase was subsequently tuned via annealing

[4], allowing for tuning the refractive index of the hybrid material. In the field of sensors, this is extended to fabricate all-sprayed hybrid biosensors [5].

Biomaterials are often affected by humidity, e.g. CNF due to their hygroscopic properties. In addition, with all-water-based processing for organic electronics, the stability under humid environment is very important. Hence, we present results on humidity cycling of hybrid polymer-CNF electrodes and water diffusion using grazing incidence small-angle neutron scattering (GISANS) [5] and quasi-elastic neutron scattering [6].

All the above-mentioned examples highlight the necessity to elucidate the structure-processing-function-relationship. To do so, advanced in situ characterization techniques on the nano- and molecular scale are a necessity. Grazing incidence small- and wide-angle X-ray scattering and GISANS allow for following and quantifying the structure formation and changes in situ during processing and subsequent treatment, e.g. annealing or humidity cycling. This allows to directly address nanoporosity in functional films as well as imbibition effects with hybrid material formation during coating for refractive index tuning.

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In situ GISAXS on cellulose-water interaction

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The determination of the detailed interfacial structure on a nano- to microscale between different materials is a significant challenge, especially for complex material systems such as cellulose-rich materials, which are ubiquitous in our daily lives. Despite the large practical importance of both living plants and cellulose-based materials, the detailed interaction between the materials during the formation of the microscopic fibre/fibre joints has remained difficult to discern. The development of new model cellulose materials and new measuring techniques has opened new possibilities to solve this unresolved problem, and here, we present a straightforward approach combining micro-focusing grazing incidence small-angle X-ray scattering (μGISAXS) and atomic force microscopy (AFM) to investigate in situ the structural rearrangements of the cellulose interfaces during drying. Model gel filaments, beads, and thin films were used to mimic the contact development in macroscopic fibre/fibre interfaces during water removal.

Figure 1 illustrates the μGISAXS measurements that were performed in the ID-12 beamline at BNL. The 2D μGISAXS scattering pattern was integrated as illustrated in Figure 1c to extract the horizontal and vertical cuts. The integrated cuts were analysed and fitted with a Guinier-Porod model [2]. Although this is a simplified approach, it is reliable enough to indicate how the structure of this complex system changes.

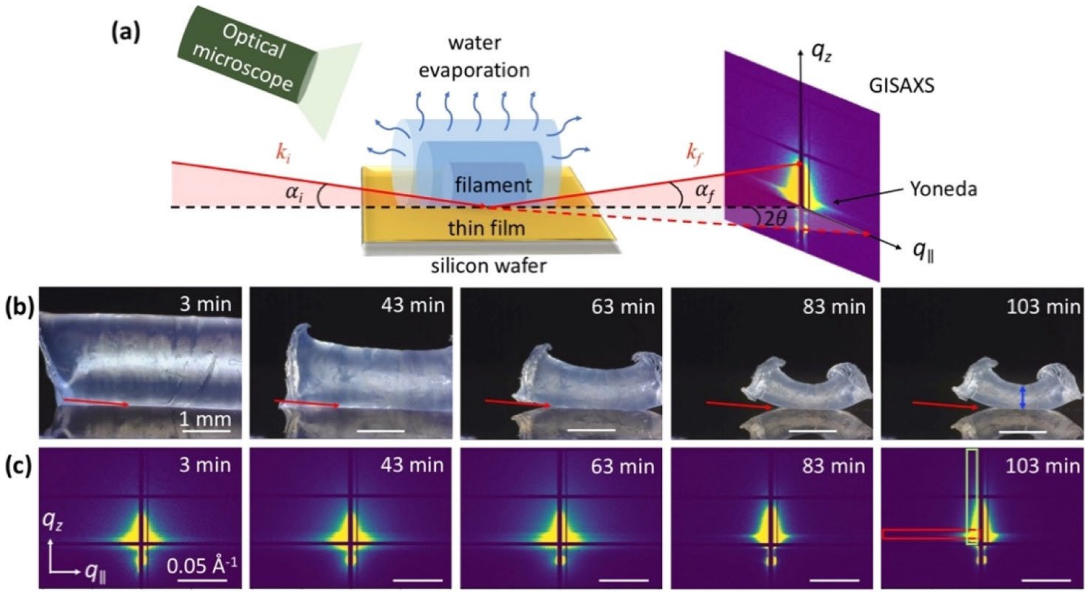

Figure 1: (a) Schematic illustration of the in situ μGISAXS measurements during drying of the water-swollen filament on a cellulose surface. $q_\parallel$ and q_z are the components of the wavevector transfer parallel and vertical to the sample surface, respectively (details can be found in the Methods section). An optical microscope was also used to monitor the filament's size during drying. (b) Representative microscope images of the cellulose filament were collected after different drying times. The red arrows indicate the incident X-ray direction. The scale bar corresponds to 1.0 mm. (c) 2D μGISAXS patterns of the interface between the cellulose filament and cellulose thin film at the corresponding evaporation time shown in (b). The red and green boxes represent the areas that were integrated for extracting the horizontal and vertical cuts, respectively. The scale bar corresponds to 0.05 $Å^{-1}$. Adopted from [1]

Additionally, we have also used SAXS to evaluate the drying of the used cellulose model material [3], as shown in Figure 2. From these measurements, it can be observed that the drying behaviour of the model cellulose material depends on the solvent and the origin of the cellulose concentration used to form it. This background information on bulk cellulose drying mechanisms was valuable in further revealing the mechanisms of the drying cellulose/cellulose interphase.

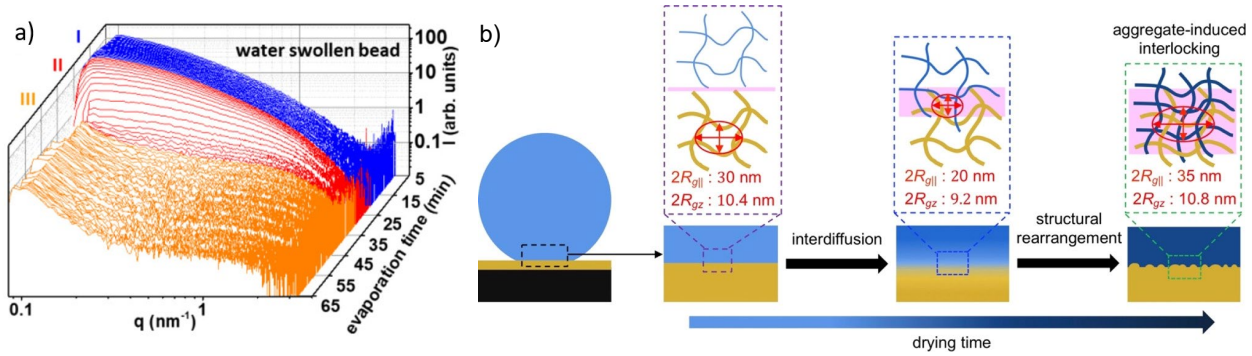

Figure 2: a) SAXS data set for drying of a water swollen cellulose bead over a time span of 60 min with a time step between each curve of 30 s, adopted from [3]. b) Postulated mechanism behind the development of the adhesive interaction between the cellulose filament and the thin film with the corresponding structural evolution of the interphase during drying, adopted from [1].

Based on the results from high-resolution measurements, including GISAXS, AFM, and SEM, we propose that molecular interdiffusion at the interface plays a major role in the development of the properties of the cellulose/cellulose interphase, which represents the development of the properties of the macroscopic fibre/fibre joints (see Figure 2). This result offers opportunities not only to understand the physics behind the fibre/fibre joint but also to facilitate fabrication and improve the properties of the composite based on these biobased soft-matter materials.

Intrigued by these µGISAXS measurements, we have started to investigate the build-up of a multi-layered structure of cellulose nanofibrils (CNF). Such layer-by-layer modified surfaces with CNF have recently [4,5] been demonstrated as an excellent platform for growing cancer cells into 3D spheroids (see Figure 3). This can be used for fundamental research on cancer mechanisms and for drug screening in the pursuit of personalised medicine. These CNF coatings work excellently; however, the reason for these cell-growth mechanisms is lacking. To fully understand the mechanisms, the structure of these coatings must be evaluated. This evaluation is problematic and cannot be done by either AFM or SEM. We have initiated tests to analyse these in a microfluidic liquid cell using µGISAXS. Some of the progress in these coating characterisation measurements will be presented.

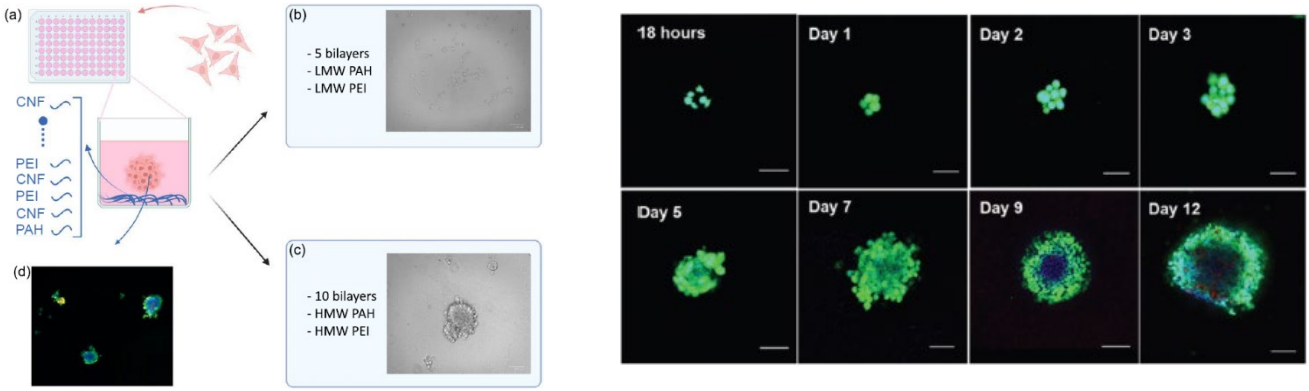

Figure 3: left) Schematic of PANC-1 spheroid formation on 5- and 10-bilayer assemblies of CNF and polyelectrolytes, adopted from [5]. right) Composite merged fluorescent images (blue colour represents Hoechst nuclear staining, green colour calcein, and red colour propidium iodide) of spheroid formation and proliferation over time at the surface of the LbL-CNF coating, scale bar (10 µm), adopted from [4].

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Challenges and Opportunities in bringing Machine Learning to Scattering

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The exponential growth in data generation at synchrotron facilities presents both unprecedented opportunities and computational challenges for the scientific community. At the Advanced Light Source (ALS), we have developed a comprehensive machine learning ecosystem that transforms how researchers interact with and analyze complex scattering and spectroscopy data. This presentation details our journey from traditional ML model deployment to the development of agentic AI frameworks.

Our computational beamline integration architecture centers on the ALS Computing Hub, which provides ML as a Service (MLaaS) capabilities directly accessible through web interfaces. We have successfully deployed unsupervised learning techniques, particularly autoencoders, for real-time denoising of spectroscopy data and dimensionality reduction through latent space visualization as shown in figure 1 and deployed at beamlines at ALS, NSLS-II and PETRA3. These tools, accessible through our MLExchange platform, enable researchers to explore complex datasets interactively, revealing hidden patterns in materials characterization that would be impossible to detect through conventional analysis.

A significant advancement in our toolkit is the development of generative AI models for X-ray scattering data. Using stable diffusion models fine-tuned on over 400,000 scattering images collected at beamline 7.3.3, we can now generate realistic synthetic scattering patterns from text descriptions or simulation parameters. This capability addresses critical challenges in training data scarcity and bias, enabling more robust model development. Our human-in-the-loop annotation system, integrated with the MLExchange Label Maker app, ensures high-quality training data while maintaining scientific validity for this development.

Beyond these established capabilities, we are beginning to explore a fundamentally different approach: the transition from traditional static model training to agentic AI frameworks. We have initiated early tests of these frameworks at the accelerator and a small tabletop beamline, taking the first steps toward systems where autonomous agents could adapt, learn, and make decisions based on evolving experimental conditions. While still in early development, these initial experiments hint at a future where AI agents might optimize data collection strategies, identify anomalies in real-time, and suggest experimental directions based on emerging patterns. This represents the beginning of a paradigm shift from "we train a model" to "agents that continuously learn and act," though significant development remains before achieving truly autonomous experimentation.

Looking forward, we envision a future where synchrotron experiments could be guided by AI agents that not only process data but understand the scientific context through knowledge graphs and ontologies.

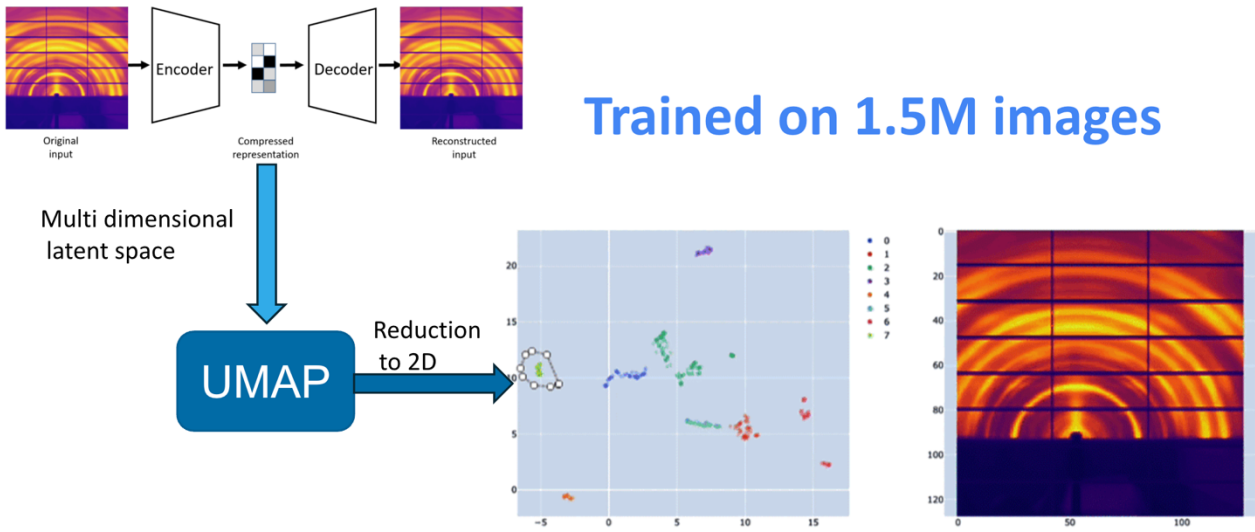

Figure 1: Development of real-time Latent Space Explorer.

Advanced GISAXS/GIWAXS characterizations for novel PV technology

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In contrast to conventional silicon-based photovoltaics, emerging thin-film solar technologies such as organic photovoltaics (OPVs), perovskite solar cells (PSCs), and quantum dot solar cells (QDSCs) offer tremendous promise for low-cost, flexible, and high-efficiency energy harvesting. Despite significant advances, novel PV technologies often suffer from dynamic structural evolution, interface instabilities, and degradation under operation. Meanwhile, these novel PV technologies rely on solution-processed functional materials whose performance is highly sensitive to their nano- and mesoscale structure. Thus, there is a growing need for non-destructive, high-resolution, time-resolved techniques capable of probing these phenomena *in-situ* and *operando* under realistic conditions. For OPVs, the phase separation between donor and acceptor domains must be precisely controlled to optimize exciton dissociation and charge transport. In PSCs, crystallization kinetics in perovskite and functional layers contribute to electronic quality and device performance. In QDSCs, ligand shell ordering and nanoparticle distribution govern charge extraction and carrier recombination.

Grazing-incidence X-ray scattering (GIXS) methods offer statistical structure and morphology information. When combined with high-brightness synchrotron sources, it enables operando studies of film evolution, nanostructure formation, and degradation under thermal, electrical, or environmental stimuli. Through case studies in OPVs, PSCs, and QDSCs, this talk will highlight how such measurements have led to fundamental insights and practical strategies for advancing device efficiency and long-term stability.

The lecture offers a few key insights and case examples about how synchrotron-based techniques help understand novel PV techniques:
OPVs: GIWAXS reveals phase distribution in the donor-acceptor film. This enabled fine-tuning of processing parameters (e.g., solid additives) to control the nanoscale structure and maximize device performance[1]. In addition, how an asymmetric non-fullerene acceptor featuring a unilateral conjugated π-bridge reduces the optical bandgap to 1.27 eV while maintaining ideal exciton dissociation and nanomorphology. Therefore, such a strategy leads to the world-record perovskite/OPV tandem solar cells[2].

PSCs: Time-resolved GIWAXS and GISAXS captured the fast crystallization dynamics of perovskite films and identified intermediate states that influence device performance. For example, the trace water in lead iodide affects perovskite crystal nucleation and results in an imbalance of charge-carrier mobilities, leading to a degradation in device performance and reduced longevity of the solar cells[3]. GIXS under environmental stress clarified how oxygen and water trigger structural collapse, leading to tailored encapsulation strategies. For example, the atmosphere-dependent degradation mechanisms for PSCs under different operation conditions. To be specific, the light-induced phase segregation, lattice shrinkage and morphology deformation occur under vacuum. Under nitrogen, only lattice shrinkage appears during the operation of solar cells, resulting in better device stability. The different behavior under nitrogen is attributed to a larger energy barrier for lattice distortion and phase segregation[4].

QDSCs: GISAXS provides insight into the inter-dot distance and the distribution which could determine the charge carrier transport of the QD solid after the deposition. By correlating nanostructure metrics with device performance, optimal interparticle spacing and cross-linking strategies were derived for improved charge transport and reduced trap states. For example, operational conditions under illumination provide a condition for spontaneously decreasing the inter-

dot distance of QDs in the active layer in combination with increasing the spatial disorder. While changes in the structure of the QDs in the hole transport layer are ruled out by further static GISAXS studies. The open-circuit voltage simultaneously decreases and exhibits a quite similar temporal evolution as that of the QD inter-dot distance in the active layer[5].

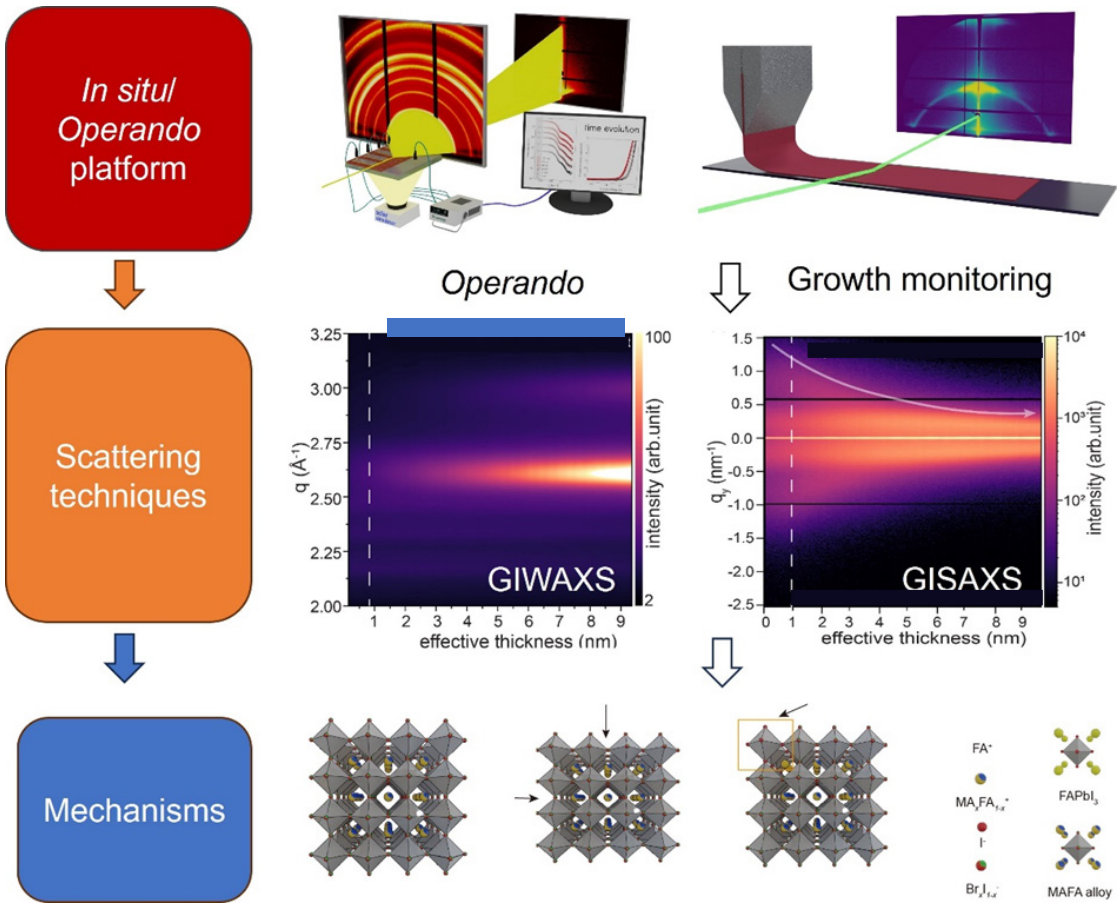

Figure 1: Schematic overview of an integrated in-situ/operando synchrotron platform for real-time monitoring of thin-film solar cell materials. The system couples GIWAXS and GISAXS with external stimuli (e.g., deposition, thermal treatment, or biasing) to probe structural evolution during film growth and operation. The GIWAXS map reveals crystalline phase formation, while the GISAXS map captures the emergence of nanoclusters and statistical domains. These insights lead to a mechanistic understanding of morphological and structural evolution in novel PV technologies.

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POSTER

POSTER

Controlled Spray coating of Bacterial Nanocellulose Thin films revealed by Surface-Sensitive X-ray Scattering

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Bacterial nanocellulose (BNC), a bioengineered polysaccharide, has emerged as a sustainable biomaterial owing to its structural purity, nanoscale fibrillar morphology, and high biocompatibility[1]. Recent research highlights the potential to tailor BNC properties by genetic engineering of producing strains or by modifying growth conditions and downstream processing. In this work, we investigate the fabrication of thin films from BNC extracted from *E.coli, S. typhimurium, Komagatereibacter,* and *Klebsiella* using airbrush spray deposition of aqueous dispersions as a green and reproducible deposition method. A particular focus is placed on how different extraction procedures (acidic, alkaline, surfactant-based) influence the nanoscale organization and adaptability of the resulting thin films. High-resolution surface-sensitive X-ray scattering techniques (μGIUSAXS/ μGIWAXS) are employed to characterize fibril orientation, aggregation behavior, and hierarchical ordering in the thin film morphology. Our findings reveal extraction-dependent structural differences that directly affect the homogeneity and functional performance of BNC thin films. By establishing optimized protocols for spray coating and scattering analysis, this study provides a versatile platform for linking chemical treatment history to structural adaptability, thereby advancing sustainable strategies for controlled thin-film fabrication and broadening the application potential of BNC in materials science and bioengineering.

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Real-Time GISAXS Investigation of Ag Thin Film Growth on Ion-Beam-Irradiated GaSb Templates

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Metallic thin films grown on nanopatterned templates provide a useful way to engineer their structure and tailor their functional properties[1,2]. Since these templates can significantly influence the changes in the surface morphology of the film, it is essential to track the structural evolution during film deposition. For monitoring these changes in real time, the *in situ* Grazing Incidence Small Angle Scattering (GISAXS) technique can be used, which provides nanometer-scale sensitivity and quantitative information on surface evolution, ordering, and growth behavior[3].

In this study, GaSb nanocone templates have been fabricated using ion beam irradiation method. It is observed that structural growth begins with irradiation-induced surface smoothening, transitioning to nanocone formation in the next stage. Ag thin films were deposited on GaSb nanocone template, and the growth process was tracked in real time using *in situ* GISAXS at P03 PETRA III DESY synchrotron facility. The evolving scattering patterns revealed distinct growth regimes- surface replication, cluster formation, coalescence into a continuous film, and shape transition of the nanoclusters from hemi-ellipsoids to cylinders after percolation, which were further validated through simulations and modelling using the BornAgain software. These results show that the combination of ion beam nanopatterning with *in situ* GISAXS offers a powerful approach for controlling and understanding the correlation between template features and metallic thin film growth, with potential applications in plasmonics, sensing, and nanophotonics.

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GISAXS/GIWAXS investigation of CVD-grown Silicon thin-film electrodes for Lithium ion battereies

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Si-based electrodes are promising material to create the next generation Li-ion batteries. In comparison to the commercially available graphite electrodes, the Si electrodes provide significantly higher gravimetric capacity (3579 mAh/g for Si vs 370 mAh/g for graphite) [1,2], owing to the alloying mechanism through which Li^+ is stored as Li_xSi ($x \leq 3.75$). Thus, the commercialization of Si into LIB will pave way for high-energy density batteries. However, despite the promising properties of the Si electrodes, its utilization is hindered by the 300% volume change that occurs during cycling. This severe volume change generates stress within the electrode, leading to cracking of the electrode and the passivating solid electrolyte interface. While the phase transformation of Si is relatively well understood [3], the formation and evolution of the SEI, as well as the phase transformation occurring in Si anodes are less explored. A comprehensive investigation of thin-film silicon model electrodes in both pristine and cycled states was performed by utilizing both grazing-incidence small-angle X-ray scattering (GISAXS) and grazing-incidence wide-angle X-ray scattering (GIWAXS) techniques, enabling detailed analysis of interfacial and structural transformations induced by electrochemical cycling.

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Crystallite Orientation and Morphology in Few-Layer TMDs Studied by Laboratory GIWAXS

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The grazing-incidence wide-angle X-ray scattering (GIWAXS) is a conventional technique for measuring textured thin films. Here, the complementary views on laboratory GIWAXS using a microfocus X-ray source are presented, demonstrating GIWAXS in time-consuming scanning modes. The scanning GIWAXS using a rotation stage is applied to the measurement of epitaxial adjustment of the few-layer transition-metal dichalcogenide (TMD) thin film of $PtTe_2$ to the c-plane sapphire substrate. The measurement of 10-11 pole figure of $PtTe_2$ can be seen in Fig. 1a, which demonstrates the feasibility of φ-scan measurement in GIWAXS mode [1], the measurement conventionally used at X-ray diffractometers. Additionally, quasi-GIWAXS tomography (QGT) on the patterned $PtTe_2$ thin film is reported, which is a technique inspired by X-ray diffraction tomography [2]. The filtered back-projection algorithm, using a Hamming filter, is employed to reconstruct the $PtTe_2$ objects created by UV laser ablation of a few-layer $PtTe_2$ thin film (Fig. 1b).

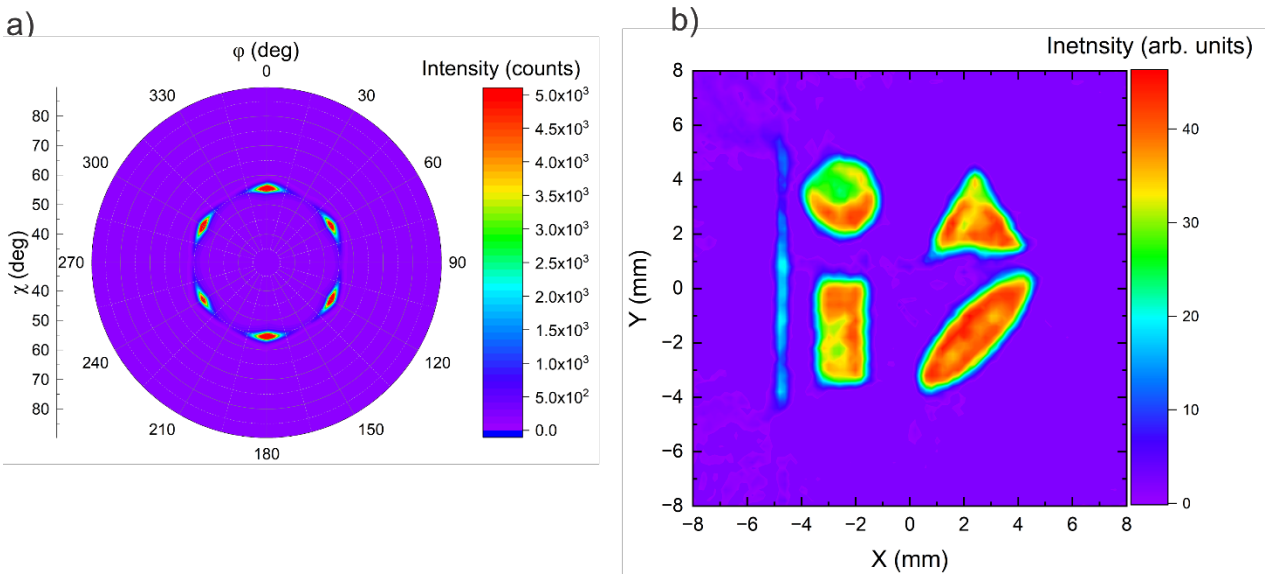

Figure 1: a) The 10-11 pole figure of $PtTe_2$ confirms in-plane hexagonal symmetry. b) The reconstructed image of the $PtTe_2$ objects based on the filtered back-projection algorithm, and measured by QGT.

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Deep Learning-Driven Grazing Incidence Small-Angle X-ray Scattering Data Processing for Nanostructure Characterization

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Understanding and controlling nanoscale architectures at surfaces and within thin films underpin advances across catalysis, energy conversion, photonics, plasmonics, and biosensing. Grazing-incidence small-angle X-ray scattering (GISAXS) noninvasively probes statistically representative areas with surface sensitivity, but quantitative analysis remains bottlenecked by model-dependent fits and the phase problem, hindering throughput and reproducibility [1].

We introduce a physics-informed, dual-branch convolutional neural network (CNN) that directly inverts 2D GISAXS patterns into (i) a discretized joint size distribution (form factor) and (ii) interparticle correlation parameters: mean center-to-center distance and positional disorder (structure factor). Training data are generated via distorted-wave Born approximation (DWBA) simulations [2] with realistic masking, noise, and preprocessing tailored to experimental conditions [3,4], enabling robust millisecond-scale inference on standard CPUs.

On synthetic validation sets, the model faithfully recovers multimodal size distributions and correlation metrics. Applied to spray-deposited Au nanoparticles, CNN-derived radius distributions agree with TEM/SEM analyses, and extracted lateral spacings are consistent with ligand shell thicknesses inferred from independent characterization. These results demonstrate that learned, nonlinear mappings can replace iterative fits for routine GISAXS workflows, offering real-time feedback for in situ studies and paving the way toward high-throughput, broadly generalizable surface nanostructure characterization.

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Biopolymer-templated Hierarchical 3D-Structured Gold Nanoparticle/Graphene Oxide Hybrid Materials for Surface-Enhanced Raman Scattering

Yingjian Guo[1,2], Guangjiu Pan[2], Suo Tu[2], Yusuf Bulut[1,2], Jungui Zhou[1], Arno Jeromin[1], Thomas F. Keller[1], Andreas Stierle[1], Gergely Nemeth[3], Ferenc Borondics[3], Benedikt Sochor[1], Sarathlal Koyiloth Vayalil[1,4], Daniel Söderberg[5], Peter Müller-Buschbaum[2] and Stephan V. Roth[1,5]

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Cellulose has emerged as a promising bio-based template for sensors, smart windows, and bioelectronics [1]. Typically, Surface-Enhanced Raman Scattering (SERS) allows for the rapid detection and structural analysis of biological and chemical compounds through their spectral patterns [2]. Crucial for SERS is fabricating substrates with strong and durable enhancements of the Raman signal over large areas and with a low fabrication cost. Herein, we present a route for fabricating cellulose nanofibril (CNF) films loaded with gold nanoparticles (Au NPs) and graphene oxide (GO) to serve as SERS substrates. All layers are fabricated subsequently by layer-by-layer spray-coating followed by annealing. Compared to two-dimensional (2D) SERS substrates, incorporating CNF substrates as three-dimensional (3D) templates ensures a more uniform distribution of Au NPs, and thermal annealing further induces more hotspots by partial sintering. Grazing incidence small-angle X-ray scattering (GISAXS) combined with nano-Fourier-transform infrared spectroscopy (nano-FTIR) was first used to study and confirm a synergistic Raman enhancement mechanism of localized surface plasmon resonance and interface charge transfer at ultrahigh spatial resolution.

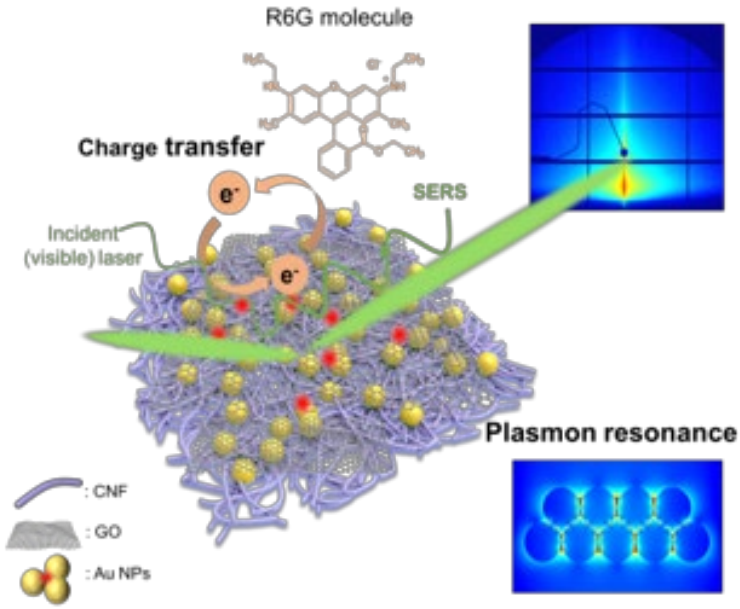

Figure 1: Schematic of our hybrid CNF/Au NPs/GO SERS platform [2].

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The kinetics of photopolymerization-induced nanostructure and interface formation for submicron additive manufacturing

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Photopolymerization offers spatial resolution, low energy consumption, and high curing speeds, making it a widely used technology in additive manufacturing [1]. The kinetics of the physical transformation of the resin from liquid to solid (cross-linked) state induced by photopolymerization and the kinetics of the solid-liquid interface formation of resin multilayer are the key to achieving controllable high-precision manufacturing. By modulating precursor resin components and combining grazing incidence small angle X-ray scattering (GISAXS), the UV-curing induced nanostructure and the kinetic of interface formation of resin multilayer are probed. We reveal how solvents and additive monomers determine the formation of nanostructures during photopolymerization and how they act simultaneously with the physical structural transformations by photopolymerization on the formation of buried interfaces in multilayer resin films.

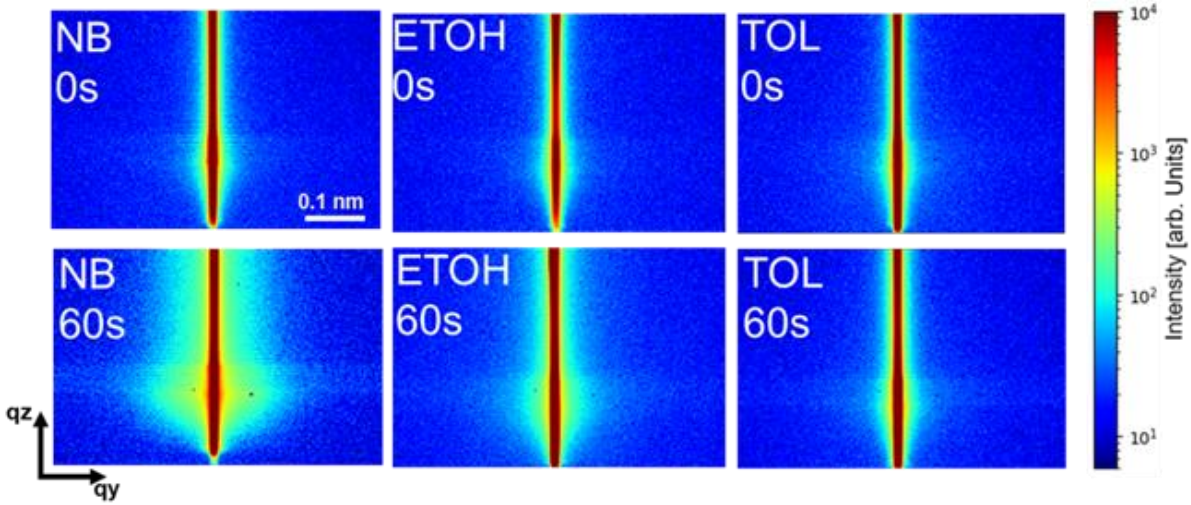

Figure 1: 2D GISAXS patterns before and after UV-cured films consisting of ethanol, n-butyl acetate and toluene as solvents.

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Vertical segregation in waterborne soft-hard binary polymer dispersions with comparable diffusion coefficients

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Binary polymeric systems (with bimodal size distribution) are widely used in many state-of-the-art materials, with applications ranging from binders for coatings and adhesives to matrixes for flexible devices. When multiple types of particles are present in a drying, continuously shrinking dispersion layer, marked differences in characteristics -such as size, diffusivity and sedimentation properties- affect the vertical profile of the resulting solid film, particularly when the rate of particle diffusion is slower compared to the rate of drying [1].

However, differences in repulsion potential can potentially also induce stratification even when the particles' diffusivity is very similar. In this study we report on the vertical segregation occurring in a system made of a mixture of hard polyacrylate and soft polyurethane colloidal particles having similar diffusion coefficients.

Through non-destructive depth-resolved ex-situ and in-situ grazing incidence small angle X-ray scattering methods, complemented by Photo-induced Force Microscopy analysis, we reveal that the vertical segregation in this system arises from a critical imbalance in the particles' behavior at larger concentration, which results in a difference in their mutual repulsion potential and leads to accumulation of polyacrylate particles at the air/film interface. Our results enrich the existing information on stratification in colloidal systems and can help to design coatings with tailored properties in the future.

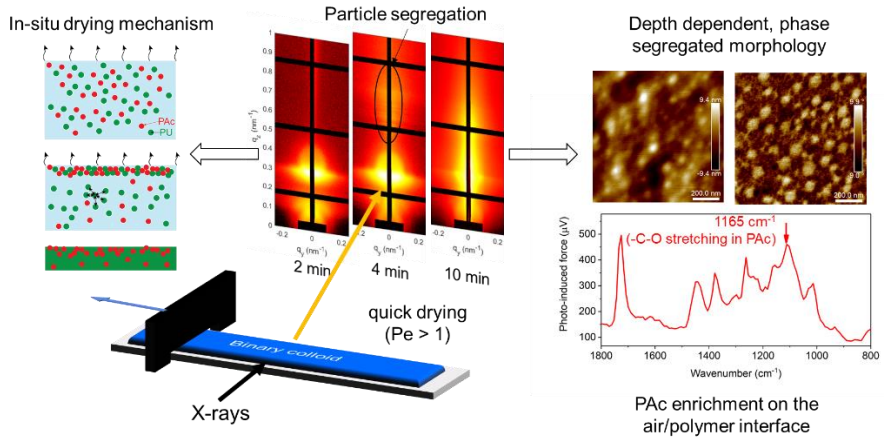

Figure 1: Graphical summary

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GISAXS of Obliquely Deposited Co_2FeAl Thin Films on Patterned Substrates

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The artificial tailoring of magnetic anisotropy by manipulating surface and interface morphology is attracting widespread interest from the application point of view in spintronic and magnetic memory devices. We present oblique angle deposition on a nano-patterned rippled substrate as a geometrical route of inducing in-plane uniaxial magnetic anisotropy (UMA) in magnetic amorphous films. For this purpose, CFA films and ripple Si substrates have been taken as a model system to demonstrate the same. Here, nano-patterned substrates are prepared by low-energy ion beam sputtering (IBS), above which films are deposited obliquely normal to the ripple directions. Grazing incidence small-angle (GISAXS) and wide X-ray scattering (GIWAXS) measurements were conducted at the P03 beamline, PETRA-III, DESY, for morphological and structural investigation. GISAXS measurements of the rippled films exhibited periodically aligned vertical streaks on either side of the specular plane (Fig.1), indicative of lateral periodicity. The asymmetric distribution of scattered intensity and the presence of inclined fringes reflect the formation of inclined columnar structures [1,2]. A clear anisotropy in the growth behavior has been observed due to the inhomogeneous in-plane organization of adatoms in the form of columns. The increased shadowing effect in the films deposited obliquely normal to the direction of the ripple patterns causes preferential coalescence of the columns along the substrate ripples, resulting in strong in-plane magnetic anisotropy in the film. This peculiarity in magnetic behavior is addressed by considering the morphological anisotropy governed by enhanced shadowing effect, the shape anisotropy, and the dipolar interactions among the magnetostatically coupled nanocolumnar structure. Our results demonstrate that deposition geometry and initial surface topography have a direct and crucial bearing on the interfacial microstructure, which can be utilized for controlled tuning of UMA by preselecting a suitable configuration.

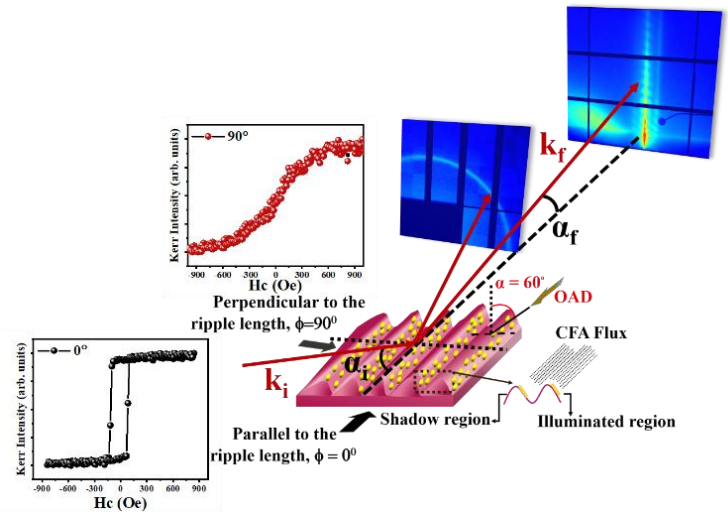

Figure 1: Schematic representation of GISAXS and GIWAXS geometry, along with MOKE loops of 50 nm CFA film deposited on rippled Si Substrates

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extending the MOUSE in spirit: toward high-quality grazing incidence in the laboratory

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We present the grazing incidence mode newly introduced at the MOUSE. Our experimental approach in X-ray scattering in transmission is extended in spirit - that means, we aim at [1]: 1) a comprehensive and well-documented methodology, 2) flexibility and wide q-range, 3) good signal-to-noise relationship, 4) traceability and uncertainty quantification, 5) data correction to absolute scale, and 6) FAIR and open data.

For thin film samples, we have to adapt MOUSE procedures in several places while elsewhere we benefit from the transmission X-ray scattering methodology. At GISAXS2025, we present our state-of-the-art, highlighting a few methodological innovations and also challenges that may need to be addressed within the GISAS community.

Our innovations address repeatability of measurements via laser-cut labels (Fig. 1, left) and a record of sample visual appearance using a toy imaging ellipsometer. Two-dimensional detector images are saved during sample alignment, allowing us to spot problems and quantify uncertainties after the fact. Using our new EPICS-empowered instrument control system we have standard measurement routines but also the option of in situ and in operando experiments as well as self-optimising measurement plans. We already optimise our beam signal-to-noise ratio and shape using Bayesian optimisation. We have successfully resolved 100 nm diameter spheres on silicon substrate (Fig 1, center) and reach $q_\perp = 16.5\,\mathrm{nm}^{-1}$ in GIWAXS using our Mo source (Fig 1, right).

Data processing is rudimentary and we see the need for joint effort in achieving absolute scale. In our view, only absolute scale will enable researchers to harness the full potential of grazing incidence scattering in the future. We look forward to this discussion, and hope that a few of our ideas inspire new and existing GISA(X)S instruments to define their philosophy and aspire to it with the full methodology in mind.

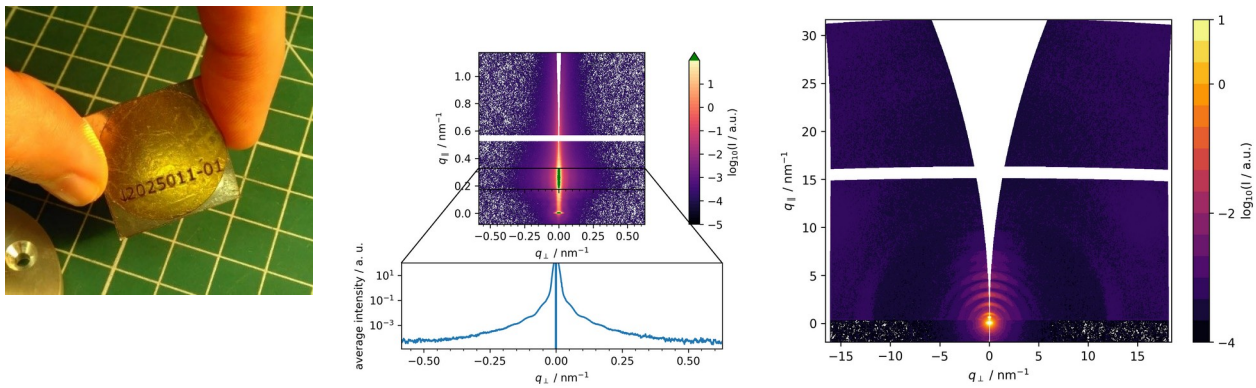

Figure 1: Left: Kapton label with id and beam direction. Center: GISAXS of 100 nm diameter polystyrene spheres on silicon, showing the sphere form factor in the line cut. Right: GIWAXS of silver behenate spin-coated onto a Si wafer, showing both the lamellar and intermolecular structure factor.

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Operando GIWAXS/GISAXS studies of SEI formation and plating process in Li/Na metal anode batteries

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Alkali metal (Li/Na) batteries are strong candidates for next-generation high-capacity energy storage due to their high theoretical specific capacities and low reduction potentials. However, uncontrolled dendrite propagation during cycling remains a key barrier to commercialization. The solid electrolyte interphase (SEI) — a 10-100 nm layer formed from electrolyte decomposition at the anode/electrolyte interface — regulates ion transport and dendrite suppression [1]. In this work, we aim to establish real-time correlations between interfacial nanoscale structural evolution and applied potential by coupling synchrotron grazing-incidence wide-angle/small-angle X-ray scattering (GIWAXS/GISAXS) with the electrochemical cycling of Li/Na battery cells.

Fig. 1 shows the results obtained during the SEI formation of 1 M $LiPF_6$ in EC:DMC. The GIWAXS pattern (Fig. 1a) reveals additional peaks identified as crystalline LiF, which formed during electrochemical cycling. Integrated intensities of LiF (111) and the amorphous peak were compared with electrochemical profiles (Fig. 1b). LiF forms mostly around 2V during small cathodic discharge, indicating electrochemical formation, while the amorphous halo grows continuously, ascribed to amorphous SEI components formation, with a step at 1V, suggesting both electrochemical and spontaneous formation. Kinks in the GISAXS pattern along Q_z (Fig. 1c) indicate a pre-existing well-defined layer that evolves during experiments.

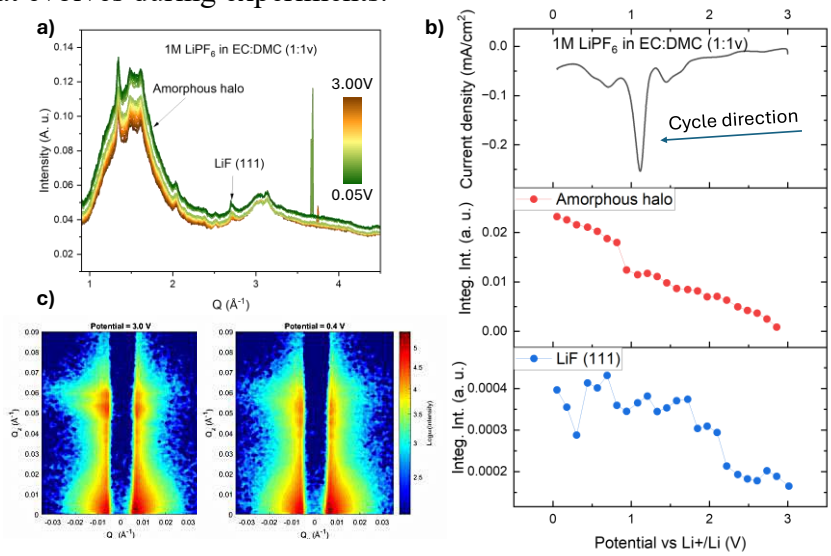

Figure 1. a)GIWAXS patterns collected during linear sweep voltammetry, b) current density profile obtained during linear sweep voltammetry (top), integrated intensities of amorphous halo (middle) and LiF (111) crystalline phase (bottom) calculated from collected GIWAXS patterns and c) GISAXS patterns collected at the start (left) and at the end (right) of the cycle for 1M $LiPF_6$ in EC:DMC.

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Acknowledgments

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Light-induced phase transitions in photoswitchable lipids

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Phase transitions in lipid assemblies are central to the functionality of lipid-based nanostructures, particularly in drug delivery systems such as lipid nanoparticles (LNPs). While endosomal release triggered by pH-induced lipid phase transitions is commonly exploited, the lack of spatiotemporal control limits its applications. In this context, photoswitchable lipids such as AzoPC offer an alternative route, since their light-induced conformational changes could, in principle, provide a driving force for structural transitions. Previous studies demonstrated that AzoPC exhibits pronounced thickness variations in vesicles [1] and significant area changes in Langmuir monolayers [2], motivating the exploration of light-induced phase transitions in bulk. To study lipid behavior in bulk, we employ grazing-incidence small-angle X-ray scattering (GISAXS) with a newly developed humidity chamber optimized for thin film studies (0.1-1um). The chamber uses a sponge-based water reservoir with dry nitrogen flow, enabling stable humidity control across a broad range. Narrow silicon wafer strips (≈1–2 mm) drastically reduce material demand, a key advantage for costly photoswitchable lipids. Reference lipids with well-characterized lattice constants serve as internal calibrants, ensuring accurate in-situ humidity determination. The compact, portable design integrates easily at synchrotron beamlines and provides optical access for light-triggered experiments.

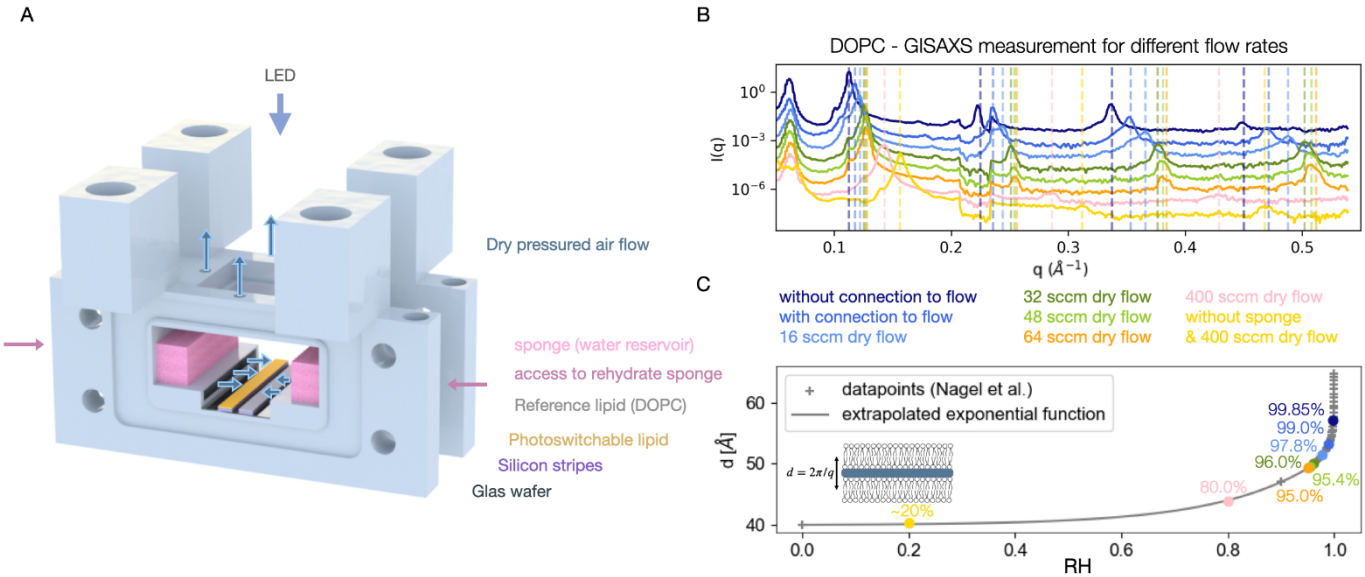

Figure 1 Humidity chamber for GISAXS experiments. A. Structure of the sponge-based humidity chamber. B. GISAXS data from DOPC thin film at different flow rates. C. Calibration of humidity using the reference lipid DOPC.

Within this framework, we will investigate AzoPC and a series of modified photoswitchable lipids with varying headgroups and photoswitch structures. By comparing their light-induced phase transitions under controlled humidity, we aim to identify the molecular parameters that govern responsiveness. The combination of minimal sample use, precise environmental control, and in-situ photo-switching provides a powerful platform to systematically explore photoswitchable lipid phase behavior and its potential in drug delivery and responsive soft materials.

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Morphological Evolution of Sputtered Ag Films on PDINN

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The controlled deposition of silver (Ag) on the organic interlayer material PDINN (perylene diimide derivative) using advanced sputtering techniques was investigated by in situ grazing-incidence small-angle X-ray scattering (GISAXS). While PDINN is widely recognized as an efficient cathode interfacial layer (CIL) in organic solar cells (OSCs), its compatibility with sputtered metal electrodes has not yet been systematically studied. In this work, we examine the morphological evolution and growth mechanisms of sputtered Ag thin films on PDINN films, with the aim of evaluating their suitability for large-scale OSC fabrication. Structural and surface analyses reveal that sputtering enables uniform, continuous Ag coverage at reduced thicknesses compared to conventional thermal evaporation, while preserving the integrity of the underlying PDINN layer [1]. These findings confirm the feasibility of sputter-deposited Ag/PDINN interfaces and establish a foundation for scalable electrode fabrication in next-generation OSCs.

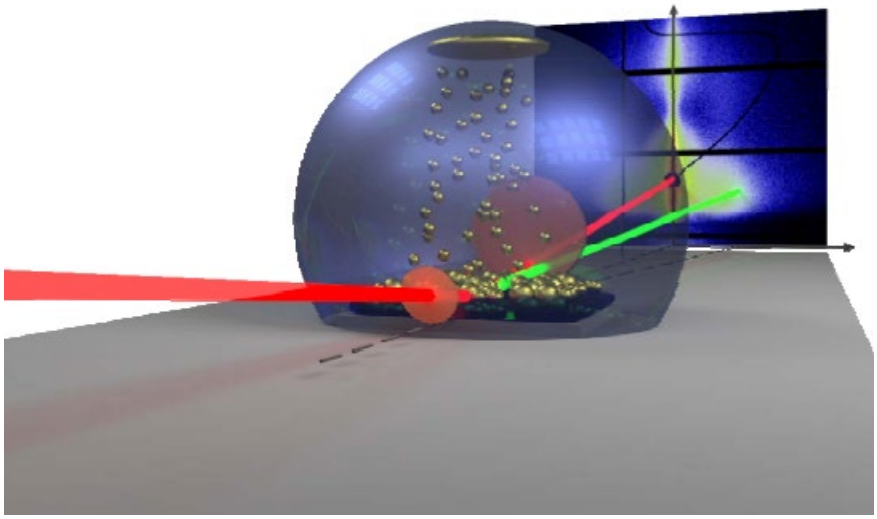

Figure 1: Schematic setup depicting in situ GISAXS measurement during the deposition of Ag on a thin-film PDINN sample in a sputter chamber

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Nanostructure Investigation of Spray Deposited Layers for Organic Solar Cells

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Organic solar cells combined with spray deposition offer cheaper, scalable, and more sustainable alternatives to inorganic solar cells[1]. To support this objective, it is crucial to understand the relationship between the fabrication process, the nanostructure of the spray deposited layers, and the functionality of the resulting device. Grazing incidence scattering techniques are ideal for investigating the morphology of the different layers and interfaces within a solar cell.
Silver nanowires are promising candidates for fabricating sprayed transparent and conductive electrodes on flexible substrates. The conductivity of the percolating network depends on both the number and quality of junctions between nanowires, factors that can be assessed using grazing incidence scattering techniques. A common strategy to improve network conductivity is to introduce a secondary material that fills the space between wires and enhances contact at wire-wire junctions. In this project, cellulose nanofibers[2] or zinc oxide nanoparticles[3] are used to fill the gaps between silver nanowires and decrease film resistance with minimal loss of transmittance.
By correlating scattering data with topographical investigations, including atomic force microscopy, and the power conversion efficiencies of full organic solar cell devices, a deeper understanding of the spraying process and nanostructure control can be achieved.

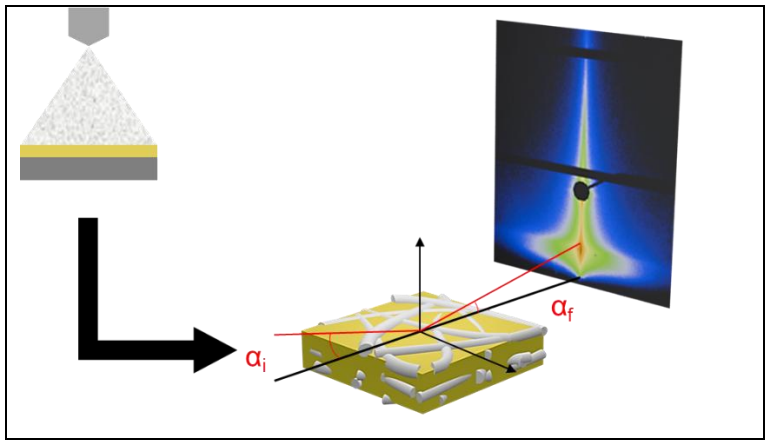

Figure 1: Spray fabrication of silver nanowire composite electrode and GISAXS characterization at a specific thickness. GISAXS image taken from Betker et al.[2].

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Vastly Increased Mesophase Range due to Superstructure Formation from In Situ Prepared Gold Nanoparticles with Liquid Crystalline Ligands: Toward Enhanced Optoelectronic Applications

Muskan[1,2], Santosh Khatavi[1], C.V. Yelamaggad[1], D.S. Shankar Rao[1], S. Krishna Prasad[1] and Bhagavatula L. V. Prasad[1,2,3*]*

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Gold nanoparticle (Au NP)-liquid crystal (LC) composites have gained considerable interest in developing cutting-edge electro-optical materials. This study introduces an innovative synthetic approach that significantly augments the capabilities of Au NP-LC composites by utilizing an amine-functionalized LC as both a reducing and capping agent in the refined Brust–Schiffrin method. In its standard form, this method yields Au NP-LC composites with appealing features like improved clearing temperature. However, the optical characteristics do not display any additional features and resemble simple LC–ligand functionalized Au NP, attributed here to the surplus LC employed to obtain the dual capabilities of reduction and capping. Thus, it was gratifying to observe a dramatic enhancement in mesophase stability with the removal of just a few percentages of excess LC, which is attributed to a remarkable and unprecedented one-dimensional superstructure formation. Furthermore, the removal of surplus LC caused the composite to exhibit Fano-like resonance in the UV–vis spectrum, a noteworthy optical feature likely resulting from dynamic plasmonic interactions, including plasmon–polariton interactions and lattice plasmon modes. Thus, the simple yet robust protocol employed yields Au NP-LC composites with an ultrawide thermal range of the mesophase that paves the way for realizing next-generation electro-optic materials featuring enhanced performance and response[1]

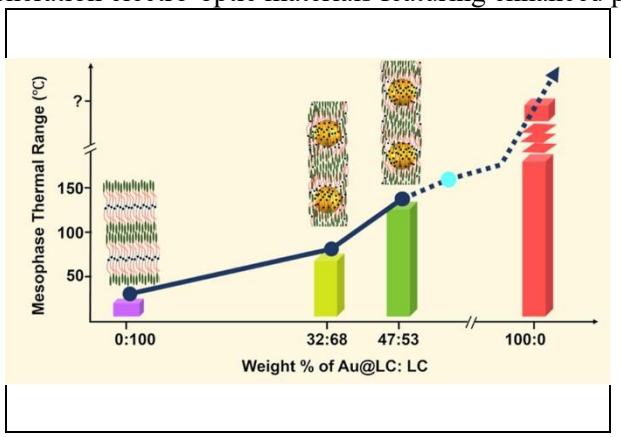

Figure 1: Schematic illustrates the extended mesophase thermal range of gold nanoparticles capped liquid crystalline ligands

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In Situ GISAXS During Sputter Deposition of Ag on a Zwitterionic Polymer Film

G. Panagiotidis[1], C. Lindenmeir[1], G. Leone[2], L. Li[1], L. Cheng[1], D. D. K. Le[1], Y. Zhai[3], K. A. Reck[4], B. Sochor[3], M. Schwartzkopf[3], A. Keloth[5], F. Faupel[4], S. K. Vayalil[3,5], T. Emrick[2], T. P. Russell[2,6], S. V. Roth[3,7], and P. Müller-Buschbaum[1]

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While organic electronic devices mainly use organic or organic-inorganic hybrid materials, many electrodes still rely on pure metals due to their unparalleled electrical conductivity. Thus, the polymer-metal interface is crucial for the overall device performance. Studies have demonstrated and established the use of ultrathin metal electrodes for (semi)transparent organic solar cells. The quality of the interface influences the formation of structural defects, which can increase resistance and the likelihood of a short circuit. For research purposes, these metal contacts are typically deposited through lab-scale methods such as thermal evaporation, while industrial manufacturing prefers large-scale methods like sputter deposition [1,2]. Additionally, zwitterionic polymers have been shown to reduce the work function of metals [3], making them promising candidates as interlayer materials in organic electronic devices. For instance, in inverted organic solar cells, these polymers can serve as interlayers between the electron-blocking layer and the silver electrode. Therefore, understanding the growth behavior of sputtered silver on zwitterionic polymer surfaces is critical. This study investigates the formation of silver electrodes during sputtering using *in situ* grazing incidence small-angle X-ray scattering (GISAXS) (Fig. 1) and atomic force microscopy (AFM).

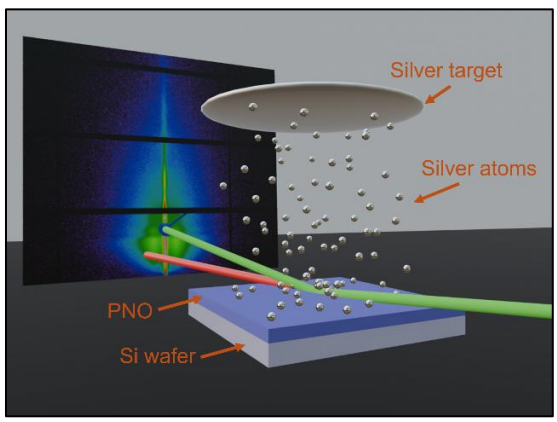

Figure 1: Schematic illustration of *in situ* GISAXS during Ag sputtering on a zwitterionic polymer film.

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In situ and ex situ solvent vapour annealing studies using GISAXS at a lab x-ray source

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We present an updated version of our portable solvothermal vapour annealing (STVA) setup [1]. The STVA chamber is smaller than in the previous version, allowing for up to 10 times faster equilibration times when filling and emptying the chamber. The design is modular, allowing 'sample table drawers' for different purposes to be slotted into the main chamber. A drawer allowing an array of permanent magnets to be mounted gives the option of annealing in the presence of a ~ 100 mT magnetic field, either in-plane or perpendicular to the thin film sample surface. The temperature is maintained via a water-based heating and cooling loop supplemented by resistive elements, and the solvent vapour environment is regulated using a commercial controlled mixing and evaporation unit.

We present examples of the use of the setup for both *in situ* and *ex situ* annealing studies of homopolymer and block copolymer thin films, together with hybrid polymer/magnetic nanoparticle thin films. The data presented are measured at RUCSAXS (Roskilde University Interdisciplinary X-ray Scattering Hub), a Xenocs Xeuss 3.0 UHR XL instrument equipped with an Eiger 2R 1 M detector and a GeniX 3D Cu- Kα HFVL X-ray tube.

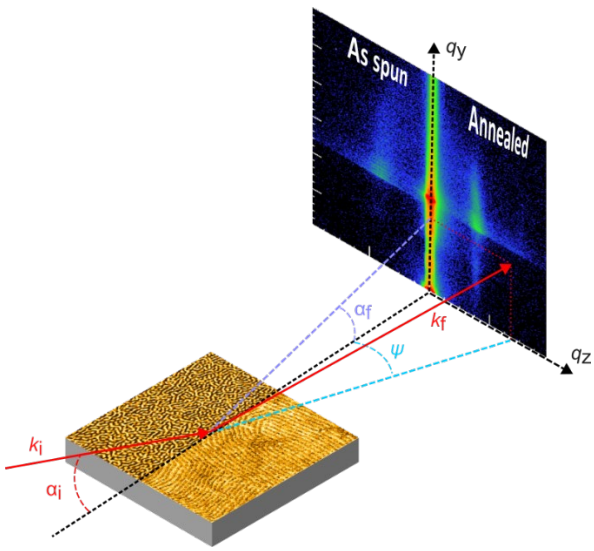

Figure 1:AFM images and corresponding GISAXS maps for a 100 nm thin film of Polystyrene-b-Polybutadiene before and after annealing in toluene vapour.

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Effect of Substrate Temperature on the Structural and Magnetic Properties of Cobalt Ferrite Thin Films

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Ferrite materials such as cobalt ferrite ($CoFe_2O_4$) are highly important for spintronic applications, including magnetic random-access memory (MRAM)[1]. To investigate the influence of thermal conditions on structural and magnetic properties, thin films of $CoFe_2O_4$ were successfully deposited on Si substrates using pulsed laser deposition (PLD) at varying substrate temperatures. Structural analyses using XRD and AFM revealed that higher deposition temperatures significantly enhance crystallinity and promote grain growth, leading to smoother surface morphologies. XPS and XAS studies confirmed temperature-driven cation redistribution between octahedral and tetrahedral sites, reflecting modifications in the electronic environment and local bonding. Crucial nanostructural information was obtained from GISAXS measurements carried out at beamline P03, PETRA-III, DESY, Hamburg, Germany. These measurements provided direct insights into nanoscale ordering and interface roughness (Fig. 1a,b). As shown in Figure 1(c), the pristine film deposited at room temperature exhibited a broad satellite peak, indicating poor ordering, island-like grains, and a symmetric crystalline arrangement. In contrast, the film deposited at 400 °C displayed sharper satellite peaks shifted towards lower qy, corresponding to increased inter-particle spacing ($d = 2\pi/q_y$) and an extended in-plane correlation length. This evolution reflects thermally induced grain coarsening, characterized by the merging of small grains into larger ones, the deformation of island-like grains, enhanced lateral ordering, and smoother interfaces—consistent with AFM observations. Magnetic characterization via SQUID-VSM and XMCD revealed enhanced magnetic moments in films deposited at elevated temperatures, consistent with the predictions of the Néel superlattice model. Overall, these findings demonstrate that substrate temperature plays a decisive role in tuning crystallinity, grain coarsening, cation distribution, nanoscale morphology, and ultimately the magnetic functionality of $CoFe_2O_4$ thin films.

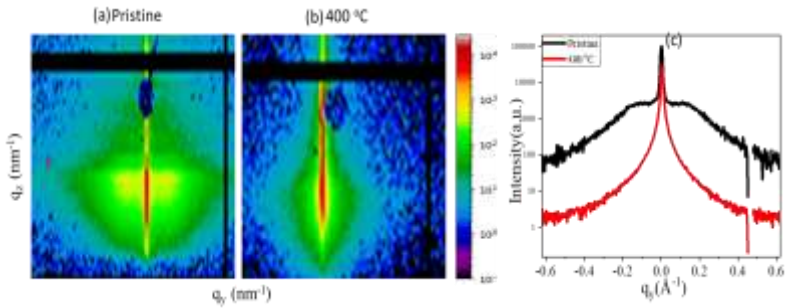

Figure 1. GISAXS of $CoFe_2O_4$ films: (a) pristine shows broad symmetric peaks, (b) 400 °C film shows sharper Yoneda region, (c) 1D I vs (q_y) profiles confirm peak shift and improved ordering.

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GISAXS Insights into Interfacial Control of Magnetic anisotropy in Zigzag Nanostructured thin films

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The engineering of magnetic anisotropy (MA) in thin films through artificial nanostructures or surface/interface micromorphology has attracted significant attention in magnetism. In the present study, we employed Sequential Oblique Angle Deposition (S-OAD) to fabricate multilayer zigzag nanostructures, which leads to an interface-induced strong MA. To date, the conventional OAD method is known to induce shape anisotropy in thin films by creating tilted columns that align the magnetic easy axis either along or perpendicular to the projection of columns. However, a key limitation of OAD lies in column merging at higher thicknesses or post-annealing, which weakens the MA [1]. In contrast, the S-OAD approach enables precise control over the number of bilayers and individual layer thickness, producing multilayer zigzag nanostructures that preserve distinct column morphology throughout the film and effectively suppress column merging [2]. Magneto-optical Kerr effect (MOKE) measurements show that cobalt and Co_2FeAl (CFA) zigzag films exhibit substantially stronger MA compared to their conventional OAD film counterparts. To explore the reason behind this MA enhancement, we have performed synchrotron-based grazing-incidence small-angle and wide-angle X-ray scattering (GISAXS and GIWAXS) measurements on zigzag and OAD samples at MiNaXS Beamline P03, PETRA III, DESY. GISAXS images of zigzag structures shown in Fig. 1 (b) reveal symmetrical horizontal streaks and vertical periodicity, indicating well-ordered alternating column tilts. The vertical line profile [Fig. 1 (e)] shows Kiessig-like fringes and a Bragg-like feature (both absent in standard OAD films), signifying conformal growth of the film due to a periodic and alternate tilt of the column in the successive layers of the zigzag sample. The origin of the Bragg-like peak lies in the periodic electron density contrasts introduced by alternating layers of tilted columns oriented in opposite directions, giving rise to the dense interfaces at their junction. These dense interface regions can act as magnetic entities that induce a shape anisotropy component perpendicular to the projection of the columns. The S-OAD method can synergize the dipolar interactions, magneto-crystalline and shape anisotropies in a single direction while preventing column coalescence, which highlights interface engineering in zigzag nanostructures as a promising strategy to tailor magnetic properties for advanced spintronic and magnetic sensing applications.

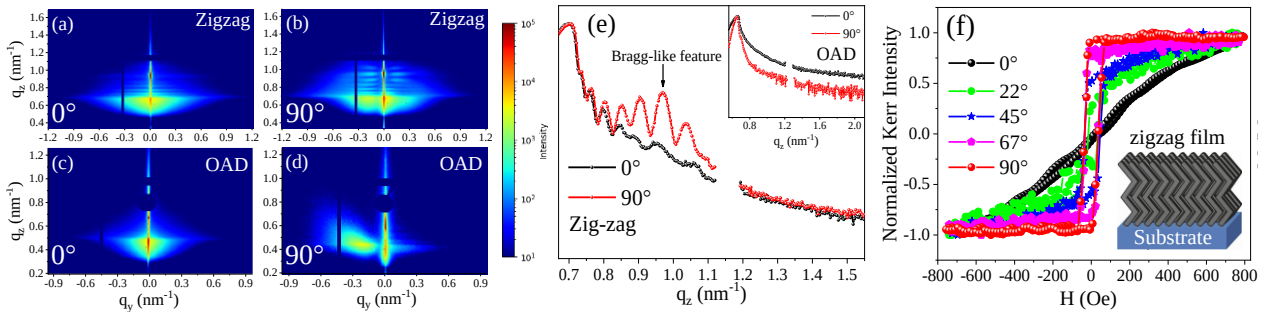

Figure 1: (a-d) shows GISAXS images of the zigzag and conventional OAD samples along (ϕ = 0°) and perpendicular (ϕ = 90°) to the projection of columns. (e) shows the vertical line profiles of the zigzag sample and conventional OAD samples. (g) displays the magnetic anisotropy probed by MOKE loops of the CFA zig-zag sample along different azimuthal directions. A schematic of zigzag sample structure is shown in the inset.

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Magnetic chirality in F/S thin films: insight via polarized GISANS

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Polarization-analyzed Grazing-Incidence-Small-Angle-Scattering (PA-GISANS) is a powerful tool to determine complex magnetic structures in ferromagnetic thin films on the mesoscopic length scale. An intriguing example are coupled ferromagnetic/superconductor (F/S) hybrid systems, where proximity effects at the interface induce exotic new quantum states. Domains and domain walls (DW's) can be used to spatially confine superconductivity [1], and with a well-defined sense of rotation of the magnetization, they are relevant for advancing the recent field of chiral spintronics [2]. Moreover, an inverse effect on the ferromagnetic state by the onset of superconductivity in systems with chiral magnetic textures is possible and can lead to a broadening of the magnetic domain walls while transiting through the superconducting critical temperature T_c (see Fig. 1). To understand these phenomena, studying the chiral magnetic textures at the F/S interface is essential, but experimental observations are challenging [3,4]. PA-GISANS provides access to both the lateral and depth-dependent magnetic structure. Especially in combination with complementary magnetic X-ray scattering techniques, such as Circular-Dichroism X-ray Resonant Magnetic Scattering (CD-XRMS), it enables a comprehensive investigation of the superconducting and magnetic properties, which we showcase for the F/S system FePd/Nb.

However, a major challenge remains in the data analysis procedures, which often rely on in-house software, developed by different research groups. A key future goal is therefore to improve and unify the data analysis protocols for PA-GISANS measurements.

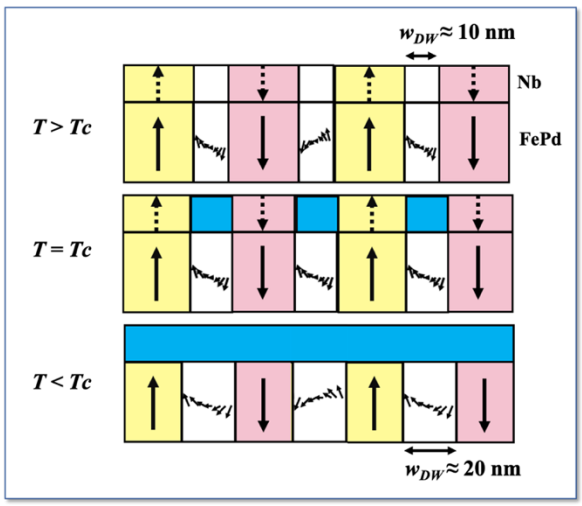

Figure 1: Change of domain wall thickness w_{DW} inside the ferromagnetic FePd layer with decreasing temperature through T_c.

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In-Situ GISAXS Tracking the Evolution of Spray-Coated Films in Organic Photovoltaics

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Ultrasonic spray deposition (USD) is a promising technique for achieving uniform material deposition on a variety of organic thin film fabrication [1]. This method holds significant potential for interfacial optimization, which is crucial for enhancing the performance and fostering the industrial scalability of organic solar cells (OSCs). In this study, in-situ Grazing Incidence Small Angle X-ray Scattering (GISAXS) is employed to investigate the film formation process of PEDOT:PSS during ultrasonic spray coating. Real-time GISAXS monitoring provides valuable insights into the evolution of nanoscale structures for PEDOT and PSS, such as phase separation and crystal size [2]. The influence of flow rate and substrate temperature on the morphology is analyzed. This work highlights how in-situ GISAXS can guide the optimization of spray coating parameters to achieve high-performance OSCs, thus advancing the scalability and efficiency of organic solar cell production.

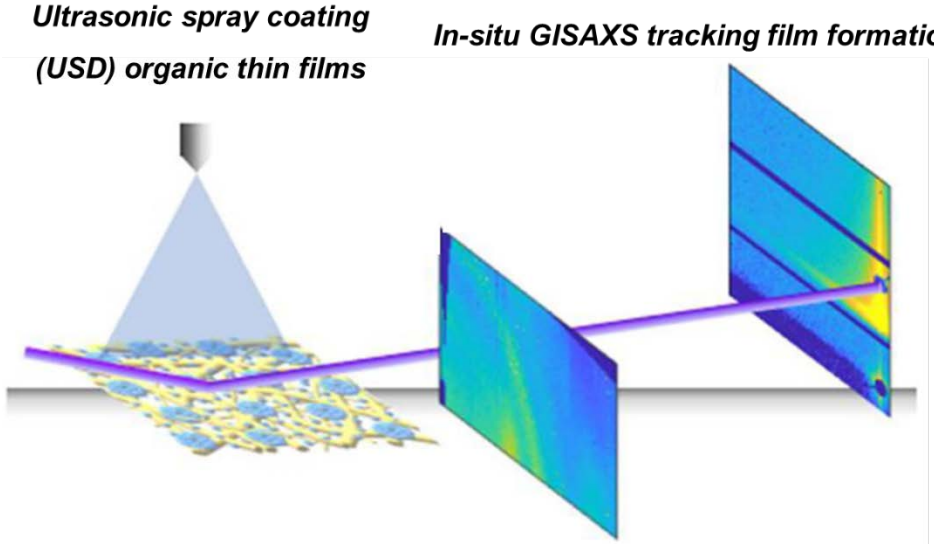

Figure 1: Ultrasonic spray deposition (USD) and in-situ GISAXS tracking film formation.

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Tiny Intrinsically Microporous Polymer Additives Enhanced Silicon-Based Lithium-Ion Batteries

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Silicon-carbon (Si/C) composite anodes are promising candidates for high-energy-density lithium-ion batteries due to the high theoretical capacity of silicon. However, their practical application is hindered by severe volume expansion and unstable solid electrolyte interphase (SEI) formation during cycling [1,2]. Fluoroethylene carbonate (FEC) is commonly used to enhance interfacial stability; however, its effectiveness is limited by concentration sensitivity and uncontrollable side reactions [3,4]. In this study, a carboxyl-functionalized polymer of intrinsic microporosity (PIM-COOH) is introduced as an electrode additive to address these challenges. The intrinsic microporous structure of PIM-COOH facilitates lithium-ion transport, while its chemical functionality promotes the preferential reduction of FEC, leading to the formation of a stable, lithium fluoride (LiF)-rich SEI layer (Figure 1). This dual effect significantly enhances the cycling stability and electrochemical performance of Si/C anodes. Notably, the Si/C@PIM||Li half-cell retains 90% of its capacity after 300 cycles at 1.0 C, demonstrating excellent long-term stability. Furthermore, when paired with a 4.5 V $LiCoO_2$ cathode, the PIM-COOH-modified anode exhibits improved interfacial compatibility and high-voltage stability. In situ grazing incidence wide-angle X-ray scattering (GIWAXS) and atomic force microscopy (AFM) analyses confirmed that the microporous framework of PIM-COOH remains intact within the polyacrylic acid (PAA) binder matrix, supporting enhanced lithium-ion transport kinetics. These results demonstrate a scalable and effective strategy for stabilizing silicon-based anodes, offering valuable insight into the design of next-generation lithium-ion batteries with both high energy density and long cycle life.

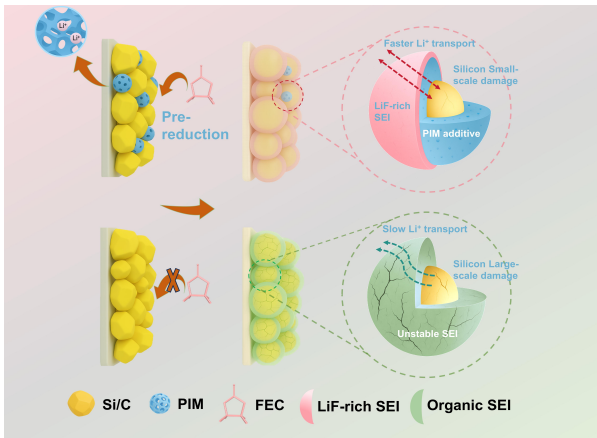

Figure 1: Mechanism diagram of the pre-reduction of FEC and the formation of SEI with different compositions in Si/C electrodes with and without PIM addition during cycling..

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Investigation of latex colloids with GISAXS

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The film formation of latex inks on nanoporous substrates is of high interest for scientific and industrial applications. Inks are multicomponent, complex fluids and consist of colloids, pigments and solvents. The film formation is a multistage process [1]. After deposition, the solvent starts to evaporate, and the latex colloidal particles begin to self-assemble leading to a close packing. Subsequently, the latex colloids undergo deformation if the substrate temperature exceeds the minimum film forming temperature (MFFT). Finally, for temperatures above the glass transition temperature, the latex colloids coalesce into a homogenous, continuous film. Despite their industrial relevance, the fundamentals of latex film formation and their observation in real-time on the nanoscale are still largely unexplored [2,3]. To quantitatively analyse latex film formation grazing incidence small-angle X-ray scattering (GISAXS) was employed on spray deposited samples at room temperature. Here, structural differences between different deposited latex colloids are visible in the scattering patterns depending on their MFFT and heat treatment. These data are accompanied by contact angle measurements, optical microscopy and scanning electron microscopy to investigate wetting and topography of the deposited layers. We present a design of an environmental spray chamber to allow in-situ GISAXS and spectral reflectance measurements during the film formation of latex colloids to be used at synchrotron facilities. Environmental conditions, i.e. temperature, humidity and air flow, will be monitored to study their influence on the latex film formation process.

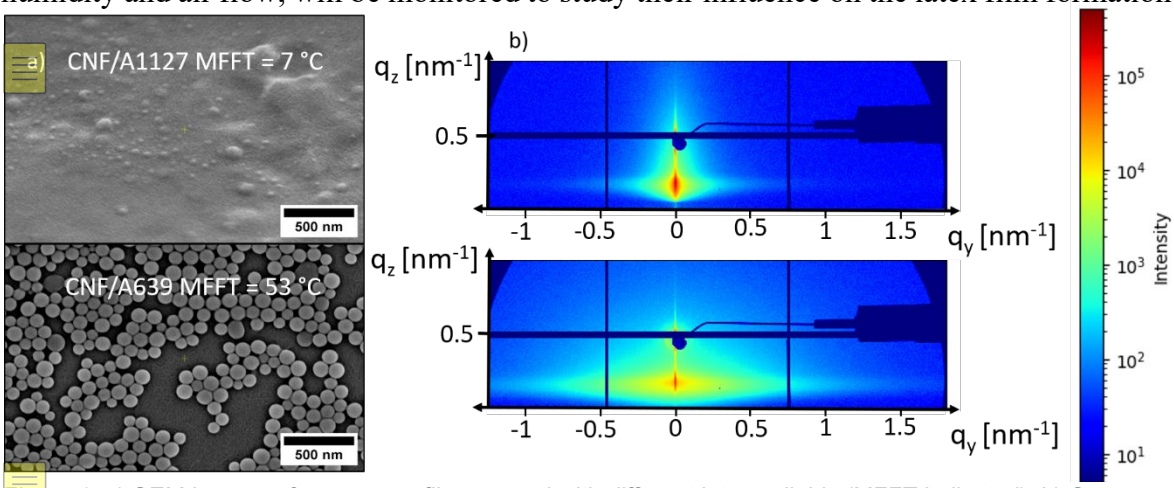

Figure 1: a) SEM images of nanopaper films coated with different latex colloids (MFFT indicated). b) Corresponding GISAXS images from ex situ measurements.

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Generalizable Physics-Informed Machine Learning for X-Ray Scattering

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Physics-informed machine learning (PIML) is increasingly applied to the analysis of small- and grazing-incidence X-ray scattering data by coupling explicit physical modeling with data-driven inference [1-3]. Embedding constraints derived from the distorted-wave Born approximation, Fresnel reflectivity, and analytic form factors within differentiable architectures enhances interpretability and computational performance relative to conventional fitting [1,2]. However, many existing implementations are tailored to specific material classes or fixed experimental geometries, which limits transferability across broader sets of nanostructures and measurement conditions [3,4].

To address this limitation, we propose a PIML framework designed to maintain physical fidelity while adapting across diverse materials and geometries. The approach combines a convolutional encoder that captures localized reciprocal-space features (e.g., fringes, rods) with a lightweight self-attention module that models long-range angular and q-space correlations. Predictions are coupled to a differentiable forward solver to ensure consistency with the distorted-wave Born approximation (DWBA) and Fresnel boundary conditions during training. The training protocol relies primarily on physics-based simulations spanning a wide range of structural and optical configurations (e.g., incident angle, wavelength, contrast, roughness, orientation distributions), while experimental datasets are used to introduce realistic detector noise, background, masking, and alignment constraints, ensuring the learned representations reflect practical beamline conditions. Representation regularization encourages latent variables to align with physically meaningful descriptors such as roughness, orientation distribution, and correlation length through auxiliary constraint terms.

The framework aims to achieve cross-material and cross-geometry transfer without retraining for each configuration, providing robust, physics-consistent inference with a clear link between data and structure. Current efforts focus on validating the method across representative scattering regimes and exploring its potential as a foundation for more transferable, physics-grounded analysis tools in X-ray scattering.

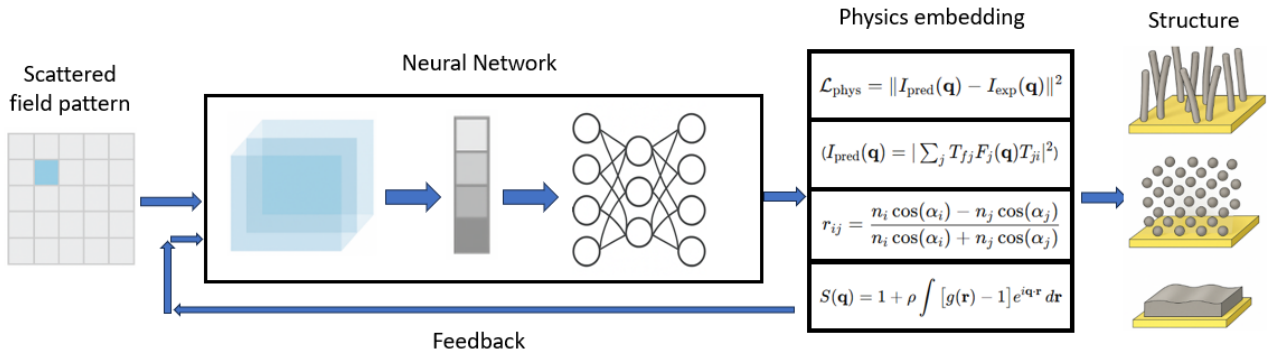

Figure 1: Workflow of the physics-informed machine-learning approach for X-ray scattering.

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