Workshop on
Coherence at ESRF-EBS

ESRF Auditorium – Grenoble, France
9 - 13 September 2019

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Workshop on
Coherence at ESRF-EBS

ESRF, Grenoble, France

ESRF auditorium

F I N A L P R O G R A M M E

Day 1 - Monday 9th September 2019

13:00 – 14:00	**Registration in the ESRF Central Building entrance hall**	
14:00 – 14:40	**Welcome/Introduction**	**B. Ruta / J. Susini**
14:40 – 16:00	**Session: Coherence: Fundamentals and EBS source** **Chair: V. Favre-Nicolin**	
14:40 – 15:20	Coherent X-rays – Fundamentals and applications	**P. Thibault (U. Southampton, UK)**
15:20 – 16:00	Coherence properties of the EBS insertion device sources	**G. Le Bec (ESRF, FR)**
16:00 – 16:30	**Coffee break**	
16:30 – 18:20	**Session: X-ray ptychography** **Chair: J. C. da Silva**	
16:30 – 17:10	X-ray ptychography: A powerful tool for imaging	**A. Diaz (PSI, CH)**
17:10 – 17:30	Ptychographic Imaging with Increasing brilliance: Strategies and developments from I13-1, Diamond Light Source	D. Batey (DLS, UK)
17:30 – 17:50	Lignin suppression in *Arabidopsis thaliana* mutant is unveiled by X-ray ptychography	C.C. Polo (LNLS, Brazil)
17:50 – 18:10	*In-situ* hard X-ray ptychography	A. Schropp (DESY, DE)
19:00 – 20:30	**Dinner at the EPN campus restaurant**	

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Day 2 - Tuesday 10th September 2019

09:00 – 10:20	**Session: Coherent diffraction imaging (CDI)** **Chair: V. Chamard**	
09:00 – 09:40	Gearing X-ray microscopy towards environmental challenges	**D. W. Breiby (Norwegian U. Sci. Tech., NO)**
09:40 – 10:00	Magnetic scattering with coherent X-ray beams: State of the art and perspectives	G. Beutier (SIMAP, FR)
10:00 – 10:20	X-ray nanodiffraction studies of PbS-nanocrystal superlattices	D. Lapkin (DESY, DE)
10:20 – 10:50	**Coffee break**	
10:50 – 12:10	**Session: Bragg CDI / Bragg ptychography** **Chair: M. Burghammer**	
10:50 – 11:30	Bragg ptychography: recent results and perspectives at fourth generation synchrotron sources	**V. Chamard (I. Fresnel, FR)**
11:30 – 11:50	What new things can we do with CDI at high coherence sources?	N. Phillips (U. of Oxford, UK)
11:50 – 12:10	Ptychographic topography in forward and Bragg direction for microscopic strain characterization	M. Verezhak (PSI, CH)
12:10 – 12:30	Plasticity at the nanoscale studied by *in-situ* nano-mechanical testing coupled with BCDI	T. Cornelius (CNRS Marseille, FR)
12:30 – 14:00	**Lunch at the EPN campus restaurant**	
14:00 – 15:40	**Session: 3D ptychography** **Chair: P. Thibault**	
14:00 – 14:40	Quantification and analysis of amorphous components in cements by synchrotron ptychographic X-ray tomography	**M. A. G. Aranda (U. Malaga, ES)**
14:40 – 15:00	Ultrafine eutectic Ti-based alloys for additive manufacturing	K. Bugelnig (DLR, DE)
15:00 – 15:20	3D morphological analysis of cement over the first 24 hours of hydration by holographic and near-field ptychographic-tomography	L. Valentini (U. di Padova, IT)
15:20 – 15:40	3D ptychography of 3rd generation solar cells	J. W. Andreasen (DTU, DK)
15:40 – 16:10	**Coffee break**	
16:10 – 17:50	**Session: X-ray phase contrast and holography** **Chair: P. Cloetens**	
16:10 – 16:50	Full-field phase imaging in practice and the impact of improved coherence	**R. Mokso (MAX IV, SE)**
16:50 – 17:10	Iterative phase retrieval in the hard X-ray tregime for absorbing multi-material objects	J. Hagemann (DESY, DE)
17:10 – 17:30	X-ray speckle-based phase-contrast imaging – State of the art and future	M.-C. Zdora (U. Southampton, UK)
17:30 – 17:50	Holographic X-ray nano-tomography to uncover neural circuits	A. Pacureanu (UCL, UK)
17:50 – 20:30	**Poster session + Cocktail**	

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Day 3 - Wednesday 11th September 2019

09:00 – 10:20	**Session: Applied Bragg CDI** **Chair: S. Leake**	
09:00 – 09:40	Bragg coherent diffractive imaging: an everyday microscopy tool?	**F. Hofmann (U. Oxford, UK)**
09:40 – 10:00	*Operando* Bragg CDI investigation of temperature hysteresis in CO oxidation on model gold catalysts	A. Passos (LNLS, BR)
10:00 – 10:20	*In situ* and *operando* structural evolution of single metallic nanoparticle model catalysts under ambient pressure reaction conditions	M.-I. Richard (CNRS Marseille, FR)
10:20 – 10:50	**Coffee break**	
10:50 – 12:30	**Session: X-ray photon correlation spectroscopy (XPCS)** **Chair: B. Ruta**	
10:50 – 11:30	X-ray photon correlation spectroscopy	**G. Grübel (DESY, DE)**
11:30 – 11:50	Complex dynamics of nanoparticles in highly concentrated entangled networks revealed by XPCS	M. Reiser (XFEL, DE)
11:50 – 12:10	Large beam X-ray photon correlation spectroscopy	F. Westermeier (DESY, DE)
12:10 – 12:30	Current progress and challenges in the imaging and control of Van der Waals high temperature superconducting heterostructures	N. Poccia (IFW Dresden, DE)
12:30 – 14:00	**Lunch at the EPN campus restaurant**	
14:00 – 15:40	**Session: SAXS XPCS** **Chair: F. Zontone**	
14:00 – 14:40	Temporal correlations in liquid water using X-rays: From seconds to femtoseconds	**F. Perakis (U. Stockholm, SE)**
14:40 – 15:00	Dynamics during an arrested protein phase separation studied by XPCS and outlook for protein-XPCS at ESRF/EBS	C. Gutt (U. Siegen, DE)
15:00 – 15:20	Heterogeneous and anisotropic dynamics during gel formation and aging of attractive colloidal suspensions	A. Jain (DESY, DE)
15:20 – 15:40	Role of higher-order spatial correlation during colloidal self-assembly	F. Lehmkühler (DESY, DE)
15:40 – 16:10	**Coffee break**	
16:10 – 16:30	Strategies to push the time resolution of X-ray photon correlation spectroscopy experiments with pixel-array detectors beyond one microsecond	E. Dufresne (ANL, US)
16:30 – 17:10	**Status ID01 / Status ID13**	
17:10 – 18:10	**Round table ID01 / ID13**	
19:30 – 22:00	**Workshop dinner**	

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Day 4 - Thursday 12th September 2019

09:00 – 10:20	**Session: WAXS XPCS** **Chair: Y. Chushkin**	
09:00 – 09:40	New opportunities to unveil glass dynamics with X-ray photon correlation spectroscopy at ESRF-EBS	**G. Baldi (U. Trento, IT)**
09:40 – 10:00	X-ray refractive parabolic axicon for coherence-related techniques: Imaging, diffraction and scattering	D. Zverev (Immanuel Kant Baltic Federal U., RU)
10:00 – 10:20	Glassy and liquid metals, from microscopic to macroscopic dynamics	E. Pineda (U. Politècnicà de Catalunya, ES)
10:20 – 10:50	**Coffee break**	
10:50 – 12:30	**Session: Applied holotomography** **Chair: J. Villanova**	
10:50 – 11:30	Holotomography for preclinical investigation in neurodegenerative diseases	**A. Cedola (Nanotec-CNR, IT*)***
11:30 – 11:50	X-ray phase contrast zoom tomography as a tool to visualize human healthy and diabetic peripheral nerves	M. Bech (Lund U., DK)
11:50 – 12:10	Correlative bio-imaging at the GINIX: Scanning SAXS, holography, and optical fluorescence	M. Osterhoff (Georg-August U. Göttingen, DE)
12:10 – 12:30	Successes and challenges of synchrotron X-ray nano-tomography for the characterization of solid oxide cells materials	M. Hubert (CEA, FR)
12:30 – 14:00	**Lunch at the EPN campus restaurant**	
14:00 – 15:40	**Session: 3D Coherent Imaging** **Chair: A. Pacureanu**	
14:00 – 14:40	Coherent diffractive imaging of non-periodic self-assembled colloidal nanocrystals	**C. Giannini (IC-CNR, IT)**
14:40 – 15:00	Internal structure of shales by coherent X-ray diffraction imaging	B. Chattopadhyay (Norwegian U. Science & Technology, NO)
15:00 – 15:20	Structural characterization of TiO_2 gig-lox sponges by coherent X-ray diffraction imaging	S. Sanzaro (CNR-IMM, IT)
15:20 – 15:40	3D analysis of bone ultra structure from magnified phase nano-CT	F. Peyrin (CREATIS, FR)
15:40 – 16:10	**Coffee break**	
16:10 – 16:30	Fast *in situ* 3D nano-imaging at the ESRF	J. Villanova (ESRF, FR)
16:30 – 17:10	**Status ID10CS / Status ID16A**	
17:10 – 18:10	**Round table ID16A / ID10CS**	
19:00 – 20:30	**Dinner at EPN campus restaurant**	

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Day 5 - Friday 13th September 2019

09:00 – 10:10	**Session: Grazing incidence XPCS** **Chair: Y. Chushkin**	
09:00 – 09:40	Future opportunities for XPCS of atomic-scale dynamics at interfaces	**B. Stephenson (ANL, USA)**
09:40 – 10:00	Liquid/liquid interface dynamics probed by grazing incidence XPCS	F. Amadei (U. Heidelberg, DE)
10:00 – 10:20	Grid-type distorted born approximation: A dynamic scattering model for coherent surface scattering imaging	M. Chu (ANL, US)
10:20 – 10:50	**Coffee break**	
	Session: EBSL1 project	
10:50 – 11:20	Project presentation	
11:20 – 12:20	**Round table EBSL1**	
12:20 – 12:30	**Conclusions**	
12:35 – 14:00	**Lunch at the EPN campus restaurant**	
14:00	End of workshop	

Abstracts

Coherent X-rays - Fundamentals and applications

P. Thibault

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Increases in X-ray source brilliance offer new opportunities for experiments that rely on X-ray coherence. This introductory talk will briefly describe fundamental concepts necessary to describe and measure the coherence properties of X-rays. An overview of the techniques that make use of coherent X-rays will then be presented.

Coherence properties of the EBS insertion device sources

G. Le Bec

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A major upgrade of the European Synchrotron Radiation Facility is in progress. This upgrade, so-called Extremely Brilliant Source (EBS), aims to increase the brightness of the photon beams by a factor of 30 or more – boosting the experiments using coherence. The augmentation of the brilliance is driven by the reduction of the size and divergence of the stored electron beam. A new electron storage ring is being installed in that purpose. The EBS storage ring and photon sources will be presented in the first part of this talk. Then, emphasis will be placed on the coherence in synchrotron radiation (SR) light sources. The tools used to describe the brilliance and the coherent fraction will be reviewed. Finally, detailed studies of a few cases will be presented, showing the properties of the EBS insertion device sources at different energies.

X-ray ptychography: A powerful tool for imaging

A. Diaz, M. Guizar-Sicairos, M. Holler, K. Wakonig, J. Ihli, M. Odstrcil, M. Verezhak, Z. Gao, D. Karpov and A. Menzel

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X-ray ptychography [1] has attracted a lot of attention in the last years because of its capability to produce images with a resolution that is not limited by lenses. This is especially appealing when using X-ray energies above a few keV, due to the challenge to fabricate high-resolution aberration-free lenses in this energy range. In fact, X-ray ptychography is much more than just a high-resolution imaging technique. Its capability to provide phase images with quantitative contrast, deconvoluted from the typically imperfect X-ray illumination, is often enough for users to choose this technique as a tool to investigate their samples due to the high quality of the images.

The implementation of X-ray ptychography is challenging due to requirements such as a coherent illumination, incident beam stability, optics-sample relative stability on the nanometer scale, and special data processing. At the cSAXS beamline at the Swiss Light Source we have developed data acquisition strategies, instrumentation and data post-processing algorithms for efficient ptychographic imaging. Our main aim is to provide a tool for users who are not familiar with the technique but can benefit enormously from it.

In this presentation we will show how our developments have successfully enabled the implementation of X-ray ptychography as a tool for users. We will show applications of in-situ 2D [2] and ex-situ 3D [3] ptychographic imaging, cryogenic imaging, [4] and high-resolution 3D imaging [5]. Furthermore, we will present our latest work towards exploiting high-brilliance synchrotron sources for even more efficient ptychographic imaging in the future, which anticipates some challenges and opportunities.

References

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Ptychographic imaging with increasing brilliance: Strategies and developments from I13-1, Diamond Light Source

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I13-1 of the Diamond Light Source (DLS) is a multiscale branchline specialised in ptychographic imaging. At 250m long, its offers a large lateral coherence length at the endstation of 100's ums. Coupled with the spacious experimental hutch and robotic detector arm, the branchline is able to operate in a variety of modes and collect multimodal data, including tomographic and Bragg ptychography.

DLS is also preparing for a potential upgrade to a diffraction limited storage ring and to make full use of the available coherent flux. I13-1 has already demonstrated the capability of producing ptychographic projections with frame times of 100us, the equivalent to scanning at 10KHz. The current hardware setup is capable of collecting ptychographic projections at 800Hz, giving an imaging rate of $200um^2s^{-1}$, with an ongoing project to push into the multi kHz regime.

Ptychographic data is processed on directly by users through *PtyREX*, a software package developed for the multimodal reconstruction and analysis of electron and X-ray data. A user interface exists in DAWN, with batch and tomography jobs being executed separately on the compute cluster. The core aim of the software is to be user friendly and effective for all experimental configurations, opening up the power of ptychographic imaging methods to novice users.

Bringing the hardware and software aspects together into a seamless process is critical for the high throughput of quality science, especially with the upcoming high brilliance sources. I will present here the latest strategies and developments from I13-1 as well as a range of use cases and results, from low contrast biological imaging to 3D Bragg ptychography.

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Lignin suppression in *Arabidopsis thaliana* mutant is unveiled by X-ray ptychography

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Lignin is a heterogeneous aromatic polymer responsible for cell wall stiffness and protection from pathogen attack[1]. However, lignin represents a bottleneck to biomass degradation due to its recalcitrance[2]. Among the different strategies to remove lignin from bagasse cell wall, the biological approach made use of genetics and molecular biology to disrupt the synthesis pathway to decrease its deposition[1], [3]–[5]. Using Ptychographic X-ray Computed Tomography (PXCT) at cSAXS beamline at SLS (PSI, Switzerland) we imaged three-dimensionally fragments of *Arabidopsis thaliana* petioles, of wild-type and C4H lignin mutant plants at cryogenic conditions. In this presentation, we will show how the 3D images revealed the heterogeneity of the cell wall morphologies along the entire cellular volume. The new results shed light the relation between disturbed lignin deposition and cellular implosion allowing to introduce a new parameter to accurately define cell implosion conditions in plants. We demonstrated how X-ray cryo-ptychography opens new insights for plant-imaging in three-dimensions to understand physiological processes.

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In-situ hard X-ray ptychography

A. Schropp, D. Brückner, X. Yang, J. Becher[1], Y. Fam[1], T. Sheppard[1], S. Weber[1], J.-D. Grunwaldt[1], and C. G. Schroer

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Hard X-ray microscopy (XRM) is a powerful and versatile characterization tool in the field of heterogeneous catalysis, allowing samples to be studied under reaction conditions in dedicated *in-situ* cells. Such *in-situ* studies are fundamental towards understanding process mechanisms and the relationship between structure and activity of catalysts. In this regard, highly spatially-resolved imaging methods have become increasingly important due to the comprehensive structural information provided. In particular, scanning coherent X-ray microscopy (ptychography) emerged as a new imaging method during the last years providing images with highest possible spatial resolution. It can be combined with fluorescence mapping (XRF), resonant scattering, tomography and, therefore, yields local structural information with elemental and chemical contrast in 2D and 3D [1-4].

In this contribution we present results of our recent efforts to build a versatile catalytic reactor cell, which is optimized for ptychographic 3D-imaging experiments under *in-situ* or *operando* conditions [5]. It enables us to carry out high-resolution imaging experiments under controlled environmental conditions of elevated temperatures and variable gas flow. Due to the enhanced stability requirements of ptychographic imaging at high spatial resolution, it is integrated into our **Pty**chographic **N**ano-**A**nalytical **Mi**croscope (PtyNAMi) at beamline P06 at PETRA III (DESY). The *in-situ* cell was designed to allow for a rotation of the sample, potentially enabling us to extend the method to limited-angle ptycho-tomography. First experiments were carried out over an angular range of about ±35°, providing 3D-structural information of catalyst samples under *in-situ* conditions.

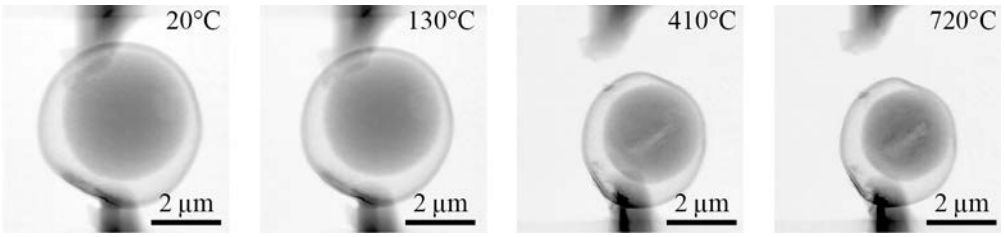

Figure 1: Series of ptychographic images of a $CoMn_2O_4$-spinel hollow sphere at varying temperatures.

In Fig. 1 an example of a ptychographic *in-situ* experiment measured on a $CoMn_2O_4$-spinel hollow sphere with 1-2 µm interior cavity is shown. The structural degredation of the sample during heating-up to 720°C is clearly visible.
Ptychographic hard X-ray imaging will considerably benefit from the high coherent X-ray flux available at the upgraded synchrotron radiation sources, such as ESRF-EBS. We aim to outline the scientific possibilities of fast *in-situ* nano-imaging at these new sources.

References

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Gearing X-ray microscopy towards environmental challenges

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These are intriguing times for X-ray microscopy as converging developments within sources, optics, sample environments, and detectors, combined with advances in artificial intelligence promise new approaches to high-performance imaging [1,2]. While sub-20 nm resolution 2D/3D X-ray microscopy is becoming an established research tool for materials science, pressing challenges await within the environmental and life sciences.
Here, we shall present our recent efforts using a selection of X-ray microscopy techniques ranging from diffraction-contrast studies of fossil bones [3], via ptychographic imaging of polymer blends for solar cells [4], to silk fibre hydration [5] – all with the aim of better understanding environmentally important porous materials. *In situ* monitoring of cement exposed to supercritical CO_2 under high temperature and pressure observed with attenuation-contrast tomography [6] will be described. Coherent X-ray diffraction imaging (CXDI) offers quantitative phase-contrast imaging of microscopic particles. Becoming able to chemically and structurally scrutinize arbitrary microscopic 3D objects ranging from algae to plastics in seawater is a prerequisite for pollution monitoring and -mitigation. Similarly, airborne particulates cause severe health and climate concerns and require statistically accurate structural models. The application of CXDI to a wide range of microparticles including Li_2ZrO_3 for CO_2 capture [7], mesoporous $CaCO_3$ [8], and metal-polymer composites [9,10] will be discussed. CXDI clearly is a unique tool for understanding mesoscale structures in both natural and manmade materials. With the rapid developments of X-ray imaging techniques, once unsurmountable microscopy challenges like deep-tissue neuronal activities, metabolism in living organisms, and multi-fluid interactions in microporous media will expectedly start receiving serious attention.

References

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Magnetic scattering with coherent X-ray beams: State of the art and perspectives

G. Beutier

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Coherent X-ray beams have been used to measure X-ray magnetic scattering at 3rd generation synchrotron sources for almost 20 years [1]. Such a combination suffers from the extreme weakness of the scattering intensity, due to both the reduced incident X-ray flux as compared to the incoherent monochromatic beam and the weak magnetic cross-section. Nevertheless, a great deal of works have been done in the soft X-ray range, where the coherent flux is higher and the magnetic scattering cross-section strongly enhanced at L absorption edges of transition metals and M absorption edges of rare-earths. Both X-ray Photon Correlation Spectroscopy [2] and imaging types of experiments have been done [3-5]. Pioneering experiments have also been done in the hard X-ray range [6-7], but the weak signal has so far prevented from going beyond the demonstration works. With a promised coherent flux greater by up to 2 orders of magnitude in the 5-15 keV range, diffraction-limited storage rings such as the EBS will provide exciting opportunities for the study of magnetism. These include antiferromagnetic orders, by using the Bragg geometry, which is often not possible with soft X-rays, and the study of micron-size 3D magnetic materials, which are opaque to soft X-rays. I will review the achievements of coherent X-ray magnetic scattering with 3rd generation synchrotron sources and detail a few perspectives.

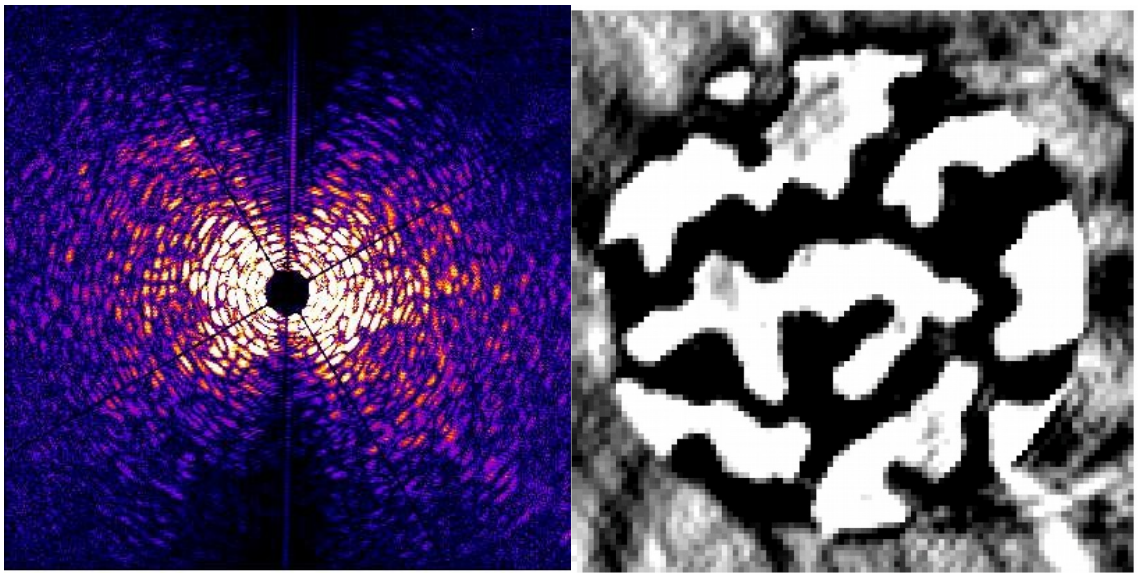

Figure 1: Magnetic speckle pattern from an holographic experiment (left) and reconstructed image of the magnetic domains (right) [Duckworth *et al*, Optics Express **19**, 16223 (2011)].

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X-ray nanodiffraction studies of PbS-nanocrystal superlattices

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X-ray nanodiffraction is applied to study the order in mesocrystalline superlattices of PbS nanocrystals (NCs) with different organic ligands – non-conductive oleic acid (OA) and semiconductive copper tetraaminophthalocyanine (Cu4APc). Anisotropic, facet-specific interactions between NCs invoke orientational order of them into mesocrystalline assemblies with a global angular correlation between the superlattice and the atomic lattices of NCs [1,2]. A detailed understanding of these correlations is expected to improve the design of NC superlattices with tailored mechanical, electric and optical properties [3].

In this work we use angular X-ray cross-correlation analysis (XCCA) [4,5] in conjunction with a nanofocused X-ray beam to reveal local correlations in the mesocrystals. The superlattices of OA-capped PbS-NCs were found to have domain structure with two different structures, which can be explained with the stabilization by different facet-to-facet interactions. Close to a grain boundary, the lattice constant decreases and the superlattice undergoes an out-of-plane rotation, while the orientation of the nanocrystals with respect to the superlattice remains unchanged. As the structural features were shown to survive upon ligand-exchange procedure [6], these results are important for the further study of properties of mesocrystals with exchanged ligands.

The Cu4APc-containing mesocrystals were grown on substrates with preliminary formed electrodes which allowed correlating their electronic properties and structure. We found dependence of the conductivity on the unit cell parameters and orientation of the superlattice. The XCCA of these samples revealed two major possible correlations between the atomic lattices and the superlattice which affect their structural and electronic properties. These results are very promising for controlling the properties of PbS-Cu4APc mesocrystals at the structure level depending on the preparation conditions.

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Bragg ptychography: recent results and perspectives at fourth generation synchrotron sources

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Ten years ago, we have proposed to develop Bragg ptychography [1] as a 3D microscopy approach dedicated to crystalline materials. These efforts were motivated by the robustness of the method with respect to highly non-homogeneous strain fields, by the high sensitivity to displacement fields, the high 3D spatial resolution and the possibility to image extended crystalline samples, pushing further away some of the limits met by finite-support based Bragg coherent diffraction imaging and transmission electron microscopy. Despite strong difficulties linked to the specific geometry of Bragg diffraction, several 3D Bragg ptychography experiments have been successfully performed on *e.g.*, biominerals at ID13-ESRF [2], phase domain boundaries at ID01-ESRF [3], stressed semiconductors at HXN-APS [4], quantum wires at HXN-NSLS2 [5], etc.

However, Bragg ptychography remains difficult to use as a routine process at synchrotrons, because it requires probe-to-sample stability over several hours combined with a fine angular exploration of the 3D intensity pattern. The inversion process involves the handling of a large 3D data set in a non-orthogonal frame, which is counter-intuitive and computing demanding.

Over the past years, strong efforts have been made at synchrotrons to promote this approach, including experimental designs and software developments. They come together with several propositions aiming at relax some of the experimental constraints, such as pre-knowledge of probe, regular angular sampling, fine angular steps, etc. Those improvements, together with the advent of fourth generation synchrotron sources, make now possible the acquisition of a full set of Bragg ptychography data in less than 30 min. These pave the way to the systematic investigation of complex crystalline systems and to the temporal follow-up of physical processes.

This talk will review the main progresses made by Bragg ptychography over these ten years and present some of the exciting perspectives offered by the advent of fourth generation synchrotron sources.

The author acknowledges funding from the European Research Council (ERC) for the European Union's Horizon H2020 research and innovation programme grant agreement No 724881.

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What new things can we do with CDI at high coherence sources?

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We will discuss some of the inherently 'new things' which might be attempted for CDI in the Bragg and zeroth-order geometry at multi-bend achromat lattice storage rings. Alongside these exciting new possibilities, we note some of the potential challenges which may require further consideration, and ways in which the community might move in order to take advantage as extremely bright sources (EBS) such as the ESRF-EBS become available.

We begin by asking the question: Does larger spatial coherence necessarily mean that a larger sample can be imaged? This is explored for both the Bragg and zeroth order geometry, and serves to highlight some approximate geometric constraints and instrument requirements for performing CDI on larger samples. Developing this line of thought, alternative scanning modalities are discussed, for example energy scanning, e-beam scanning, event mode data acquisition and more generally multi-modal data acquisition, including the feasibility of collecting X-ray and electron microscopy data in the same instrument. This includes discussing the adoption of CDI methods by traditionally non-coherence beamlines and the need not only for data pipelines, but in some cases, a simplification of the data for non-expert users in order to facilitate widespread use and acceptance of CDI methods as part of the standard microscopy toolkit.

By briefly discussing some of the potential challenges and exciting new possibilities possible at upgraded sources we hope to prompt further discussion and development of ideas for the growing CDI community at next generation synchrotrons.

Ptychographic topography in forward and Bragg direction for microscopic strain characterization

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In addition to high-resolution imaging, coherence can be exploited to study strain fields with high sensitivity. We present a technique developed at cSAXS (SLS) that allows successful imaging and reconstruction of the strain field present in a crystal, both in forward and Bragg configuration.

Scanning a pinhole placed a few millimetres downstream of the sample[1] in either the forward or the Bragg direction, and recording the far-field diffraction patterns when the sample fulfils the Bragg condition allows ptychographic reconstruction of the exit wave front at the pinhole position. Reconstructions at the sample position are then obtained after numerical backpropagation (Figure 1b), and exhibit strain sensitivity at each angular position during a rocking curve measurement.

3D Bragg ptychographic reconstructions are known to be challenging for highly strained samples with thickness above a few hundred nanometres. Using ptychographic topography, here we demonstrate imaging of strain fields inside a 2 μm diameter InSb pillar[2] after uniaxial compression.

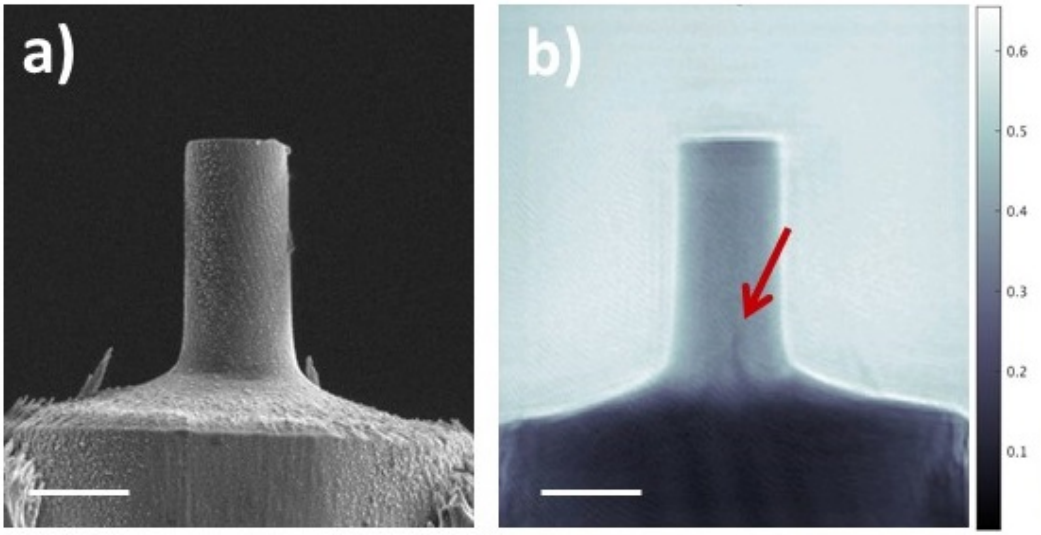

Figure 1: a) SEM micrograph of InSb micropillar after uniaxial compression, b) amplitude of ptychographic topography reconstruction in forward direction with strained region shown by red arrow. Scale bar: 2 μm.

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Plasticity at the nanoscale studied by *in-situ* nano-mechanical testing coupled with BCDI

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The mechanical properties of micro- and nanostructures were demonstrated to differ significantly from those of their bulk counterparts. Despite numerous studies, plasticity at the nanoscale is, however, not fully understood yet. In particular, the influence of surface diffusion on the defect nucleation process is still an open question. *In-situ* experiments are perfectly suited for the fundamental understanding of the onset of dislocation nucleation. Thanks to its high sensitivity to strain and defects as well as its non-invasive nature, Bragg coherent X-ray diffraction imaging (BCDI) is the ideal tool to be coupled with nano-mechanical testing devices to study plasticity at the nanoscale *in-situ*. BCDI on indented Au crystals demonstrated the capability to imaging a single prismatic loop trapped in the crystal [1]. Actual *in-situ* nano-indentation was rendered possible by scanning the energy of the incident X-ray beam instead of rocking the sample which might induce vibrations that are detrimental to the nano-mechanical setup [2]. This mutli-wavelength (mw) BCDI approach allows for imaging the evolution of strain and defects during mechanical loading as demonstrated by Fig. 1(a)–(c) [3]. The defects highlighted by an arrow in part (c) are actually dislocation loops as shown in the slice of the reconstructed phase field presented in Fig. 1(d).

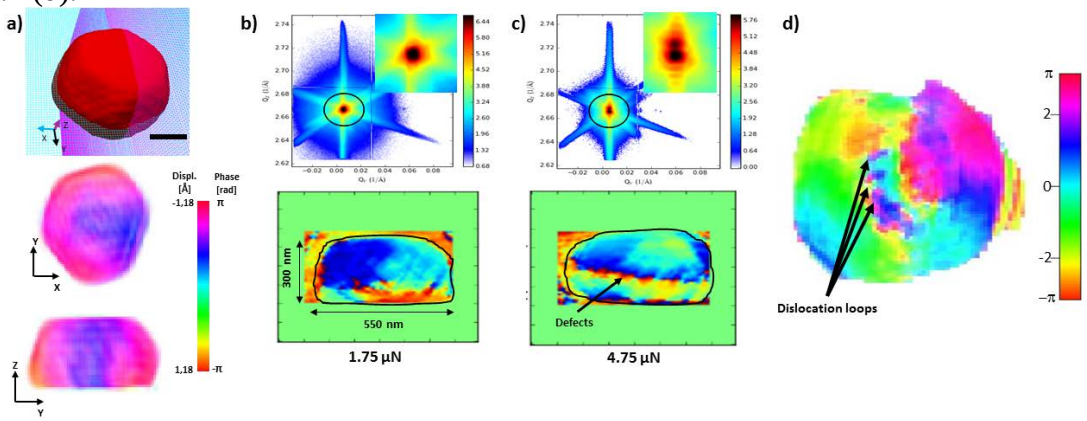

Figure 1: a) Reconstructions of both the shape and the phase of a gold crystal measured by mw-BCDI [2]. BCD pattern and reconstructed phase field for a gold crystal indented with an AFM apllying a load of b) 1.75 and c) 4.75 µN. d) Slice of the reconstructed phase filed presented in part (c) evidencing the presence of dislocation loops [3].

The tremendous increase of the coherent flux of the ESRF-EBS will boost the time resolution, eventually allowing for imaging the defect nucleation and diffusion on the timescale of seconds. In addition to allowing for measurements on shorter timescales it will also reduce the effects of drifts and any other instability. Further improvements on phase retrieval algorithms such as multiple reflection BCDI and defect analysis will bring coherent X-ray diffraction and the EBS to the next level.

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Quantification and analysis of amorphous components in cements by synchrotron ptychographic X-ray tomography

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Building materials have complex hierarchical microstructures. To fully understand their main properties a sound description of their spatially-resolved contents is compulsory. Developing this knowledge is challenging as about half of the volume is amorphous/nanocrystalline. Furthermore, more than one amorphous component can coexist which is even more challenging to quantify with standard techniques.

On the other hand, ptychographic X-ray computed tomography (PXCT) is a non-destructive X-ray imaging technique which retrieves the full refractive index of the sample, 3D electron density –rho_e– (very related to mass density) and attenuation coefficient –mu– distributions, with a 3D isotropic resolution, nowadays slightly better than 100 nm for cement pastes. Crystallinity is not required and spatial analysis can be carried out by a set of tools including segmentation of the different material phases in the 3D dataset(s). Acknowledging the intrinsic lower spatial resolution in the reconstructed absorption tomograms, the importance of having both (rho_e and mu tomograms) will be highlighted for studying complex samples with amorphous components.

Here, we will review our ongoing efforts to contribute to the understanding in cement science at the mesoscale by analysing and quantifying the amorphous component(s). This is key in cements as volume stability of buildings is key and mass density values (and their spatial distribution) must be known, which is far from trivial for amorphous components intermixed with crystalline phases.

Firstly, we will discuss our work on Portland cement [1] where three unaltered samples: neat Portland cement (PC) paste, PC-calcite and PC-fly ash blended pastes, were analysed. For the neat PC paste, the PXCT study gave densities of 2.11 and 2.52 gcm^{-3} and contents of 41.1 and 6.4 vol% for nanocrystalline calcium silicate hydrate (C-S-H) gel and poorly crystalline iron-siliceous hydrogarnet, respectively. Furthermore, it was possible to differentiate inner product and outer product C-S-H gels. Amorphous fly ash content was also analysed. Further details will be provided.

Secondly, we will elaborate on the use of PXCT for characterizing ye'elimite-based ecocements [2]. These cements are interesting as an alternative to Portland cements to decrease the CO_2 footprint of cement production. It was possible to discriminate between an aluminium hydroxide gel and calcium aluminium monosulfate, which have close electron density values. Specifically, the composition and mass density of two aluminium hydroxide amorphous gel agglomerates were determined: $(CaO)_{0.04}Al(OH)_3 \cdot 2.3H_2O$ with 1.48 gcm^{-3} and $(CaO)_{0.12}Al(OH)_3$ with 2.05 gcm^{-3}.

Finally, desired performances for post-EBS (10-30 keV) PXCT will be sketched.

Acknowledgements. This work has been supported through BIA2014-57658 and BIA2017-82391-R research grants, which are co-funded by FEDER.

Data accessibility. All reconstructed 'raw' tomograms (tiff format) were deposited, to be freely accessed, on Zenodo; dois: 10.5281/zenodo.2533863 & 10.5281/zenodo.3202557, for OPC and ye'elimite-based pastes, respectively.

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Ultrafine eutectic Ti-based alloys for additive manufacturing

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We are currently developing novel high strength alloys with ultrafine eutectic microstructures using additive manufacturing (AM) to increase the performance of turbocharger compressor wheels. The microstructures of eutectic and near-eutectic alloys display features such as dendrites and lamellar structures that can extend over several length scales [1]. The size of these features commonly decreases with increasing growth velocity or cooling rate during solidification. This gives rise to significant size strengthening effects and opens a gateway to design materials by rapid solidification. Particularly, selective laser melting (SLM) is an AM rapid solidification technologie that may be used for this objective.

For this purpose, Ti-based ultrafine eutectic alloys (Ti-32Fe, Ti-28Fe-9Sn-5Nb and Ti-21Fe-7Sn-4Nb, wt.%) were manufactured by SLM applying various pre-heat temperatures (200 - 790 °C). The microstuctures were characterized three dimensionally using near-field ptychographic tomography (PXCT) with voxel size as low as $(15\ nm)^3$ and complemented by synchrotron radiation diffraction (SXRD), scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD).

Crack free samples of the investigated alloys could be manufactured only at increased substrate temperatures (650 - 790 °C). SEM and, more importantly, PXCT revealed fine eutectic β-Ti/TiFe structures with lamellar spacings even below 100 nm as well as globular structures in the interlayer boundary located at the bottom of the melt pools. Elevated pre-heat temperatures and high energy densities favoured the formation of oxygen stabilized Ti_2Fe (also known as $Ti_4Fe_2O_x$ [2]) which appeared as dendrites within the eutectic structure (Figure 1). Mass densities of all phases derived from PXCT were in good agreement with those determined from SXRD. The high resolution 3D images and the possibility to quantify microstructures down to nm-range extend the horizon for the development of new materials tailored to the metallurgical conditions of additive manufacturing.

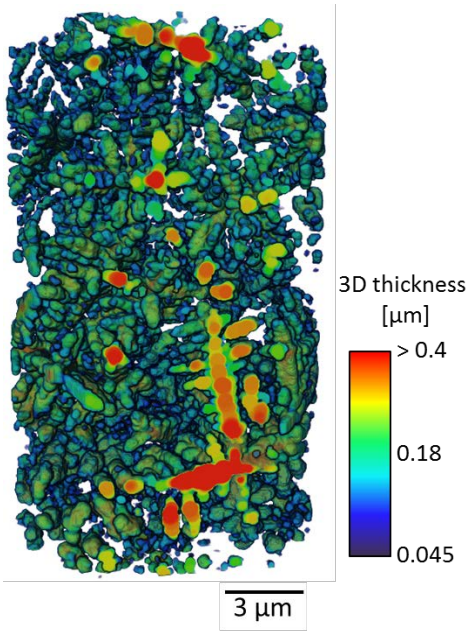

Figure 1: 3D thickness distribution of $Ti_4Fe_2O_x$ dendrites

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3D morphological analysis of cement over the first 24 hours of hydration by Holographic and Near-Field Ptychographic-tomography

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Although a very old but industrially important material, the hydration of cement is a process that is not fully understood, in particular over the first 24 hours of hydration. The main mineralogical phase in the ordinary Portland cement is the tricalcium silicate (C_3S). The hydration kinetics of the C_3S particles proceeds in different hydration stages. After an induction period with low reaction degree, the majority of C_3S reacts during the main hydration period to form C-S-H (calcium-silicate hydrate) and portlandite (calcium hydroxide). Despite the fact that the hydration kinetics is governed by the particle size distribution of the starting material, no information is available on the development of the particle size distribution of C_3S during hydration.

The present work is therefore aimed to fill this gap [1]. By the first time, the particle size distributions of C_3S during the first 24 hours of hydration were experimentally measured with sufficient high spatial resolution by Holographic and Near-field Ptychographic X-ray Computed Tomography. The measurements were performed at four point in time: 0h, 12h, 16h and 20h of hydration. The gathered results are in good agreement with the published models having the degree of hydration as main input parameter. They provide sufficiently detailed information about the modes of C_3S dissolution during cement hydration, which is very important for the understanding of the cement hydration.

In addition, the holographic and ptychographic imaging measurements showed a very good correlation. The time-dependent particle size distribution can be finally tracked with with the necessary spatial resolution using those coherent X-ray imaging techniques. This work also demonstrates the possibilities offered by the use of coherent X-rays to characterize minerals of industrial interest such as the C_3S, which will fully benefit from the upgrade source ESRF-EBS.

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3D ptychography of 3rd generation solar cells

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A large variety of chemical compounds, both inorganic and organic, constitutes the so-called 3rd generation thin-film solar cells. This includes compounds belonging to the family of perovskites and chalcogenides, polymers and organic small molecules. Common to all the thin-film solar cell technologies is the strong correlation between nano-structure of the photo-active layer, interfaces and electrodes, and the performance of the complete device.

State of the art 3D ptychography applied to a tandem polymer solar cell with a hierarchical structure covering characteristic phase separations from a few nm up to hundreds of nm, showed that a spatial resolution of about 20 nm can be achieved in 3D, albeit presumably not for the weakest material contrasts (Figure 1, left) [1].

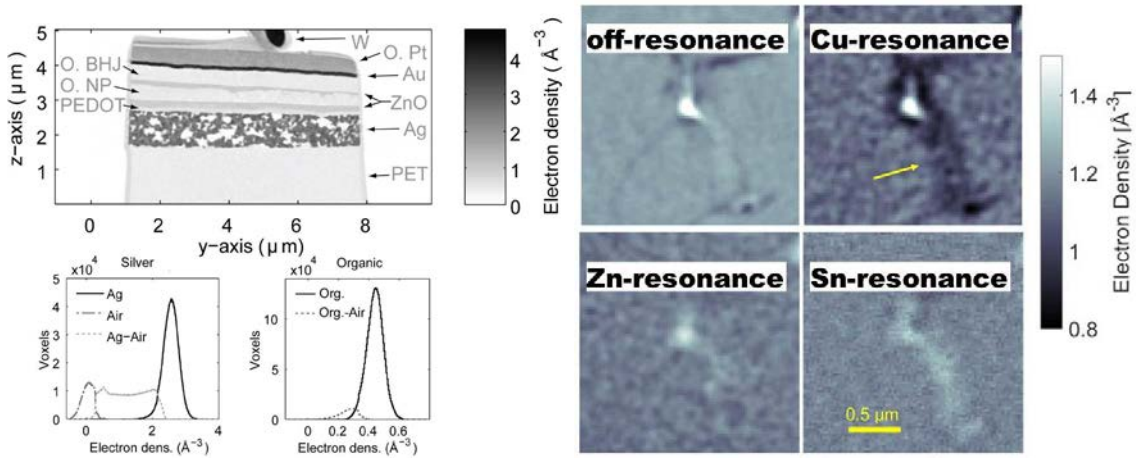

Figure 1 **Left :** Slice of tandem polymer solar cell, showing two absorber layers (BHJ + NP) between electrodes and recombination layer (ZnO), with examples of electronic density segmentation below. **Right:** Ptychographic tomogram slices of grain boundaries with impurity phases in a CZTS solar cell at 4 X-ray energies showing different contrasts as a function of concentration of the resonating elements.

With a recent example from the family of chalcogenide solar cells, we have demonstrated an unprecedented quantitative chemical sensitivity by application of resonant 3D ptychography (Figure 1, right). Interfacial defects and secondary phases of a Copper Zinc Tin Sulfide (CZTS) active layer are resolved with resonant X-ray ptychographic tomography (RXPT) [2] allowing large field of view covering an entire functional device with high spatial resolution. We find that RXPT allows us to highlight morphology of the active layer and contacts to the interfacial layers, and to map and quantify secondary phases that cannot otherwise be discriminated. The applications stand to experience substantial improvements with the higher coherent flux of 4th generation synchrotrons.

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Full-field phase imaging in practice and the impact of improved coherence

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Phase contrast imaging may be performed at various levels of sophistication. At times one is satisfied with only an edge enhanced picture of the sample, other applications require to calculate the projected density and so to say convert the edge contrast to area contrast. A number of algorithms exist for the latter, yet mainly the transport of intensity based homogeneous object type of phasing method was deployed in the past years on most imaging beamlines. This method works well on data acquired with X-rays of small coherence length and a single exposure (one defocus image). It is debated to which extend would larger coherence length result in images of higher quality. This question can be addressed from two aspects: higher quality with same scanning times or higher quality with same deposited dose. I will discuss these aspects of phase contrast imaging and demonstrate on examples ranging from lab microCT through synchrotron beamlines [1] to XFEL [2] data. Equally I will highlight recent developments on reconstruction of dynamic phase contrast tomographic data [3].

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Iterative phase retrieval in the hard X-ray regime for absorbing multi-material objects

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X-ray near-field holography (NFH) is a lens-less phase-contrast imaging technique which can be readily implemented at synchrotron imaging setups. NFH is suited to image a wide range of objects from weakly interacting to strongly absorbing multi-material specimen.
The amplitude and phase information of the specimen is encoded in the measurement by the free space propagation of the wave behind the object. Making use of a diverging beam as it can be created via different variants of focusing or waveguiding [1] a scalable field of view is achieved by changing the magnification simply via the focus to sample distance.
The aim of the current work is to extend the reconstruction process beyond the assumption of weak or coupled (δ/β ratio) specimen [2].
We have extended the iterative phase retrieval scheme developed in [1], to multi-material objects. The principle scheme of an alternating projection scheme stays the same. But by introducing ‚momentum' for an increased convergence speed and several adjustments to the sample projector P_S (e.g. applying phase unwrapping, smoothing the support) we have extended the range of application further.
We demonstrate the algorithm on an ivory sample partly covered with Gallium due to preparation process by FIB. The data shown in Fig. 1 has been acquired at the nano-branch of the beamline P05 at PETRAIII. Using both experimental hutches of P05 an overall propagation distance of 16.5 m could be achieved, yielding a magnification of ≈ 220.
NFH as scheme benefits greatly from 4th generation sources. Higher coherent flux and diffraction limited focusing will push the development of this technique further. Supplemented by improved reconstruction schemes, the material composition of a sample can be determined from the reconstruction of the complex valued wave field.

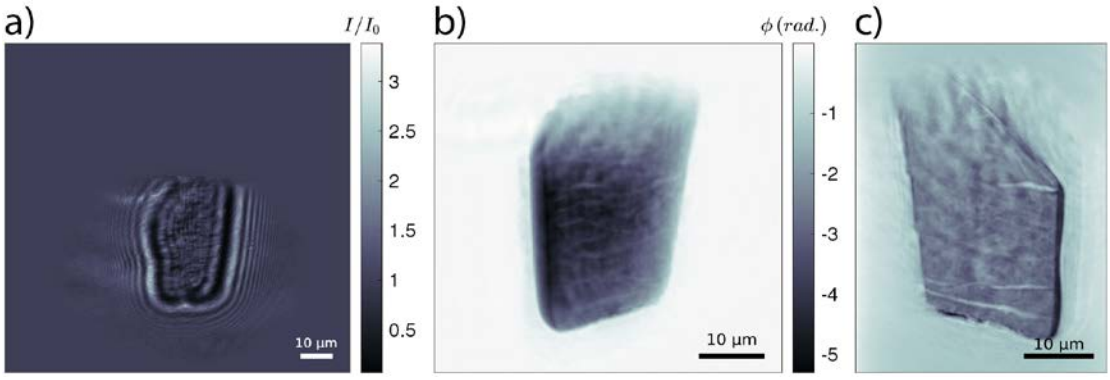

Figure 1: a) Flat-field corrected hologram at $Fr = 2 \cdot 10^{-4}$ of an ivory sample. The illumination was created by a FZP of 300 µm with outermost zone width of 100 nm, 1 s exposure time. b) Image of the phases of a reconstructed projection using the presented approach. c) Ortho slice of the reconstructed volume, showing the Gallium crust on the right deposited by ion beam milling.

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X-ray speckle-based phase-contrast imaging - State of the art and future

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X-ray speckle-based imaging (SBI) is one of the youngest full-field phase-contrast imaging methods [1,2]. In the last years, it has seen rapidly rising interest and has found applications in various fields including metrology, biomedicine, materials science and more [3].

SBI relies on the use of a near-field speckle pattern as a wavefront marker, which is created when a diffuser with randomly distributed small particles is illuminated by (partially) coherent X-rays. When a sample is introduced in the beam, its properties are encoded in modulations of the pattern. Analysis in real space delivers the differential phase signal as well as complementary information on X-ray transmission and small-angle scattering. SBI phase tomography provides 3D high-contrast structural information and moreover the quantitative electron density distribution. While the achievable spatial resolution cannot compete with other phase-sensitive methods such as ptychography, SBI benefits from a simple, robust setup and data acquisition and moderate coherence requirements.

Here, we present the principles and state of the art of SBI and point out its future potential at ESRF-EBS. We particularly focus on our recently proposed data acquisition and analysis scheme, the unified modulated pattern analysis (UMPA), which allows for a simplified scan routine and flexible tuning of angular sensitivity and spatial resolution [4]. We demonstrate the potential of UMPA for different applications such as the characterisation of X-ray refractive lenses [5], 3D virtual histology of hydrated, unstained tissue [6], geological investigations of volcanic rocks and materials science studies of mortar, see Fig. 1.

The latter are part of most recent efforts to translate SBI to higher X-ray energies. Although SBI does not impose as stringent coherence requirements as other phase-sensitive methods, speckle visibility gets poor for transverse coherence lengths much shorter than the diffuser grain size. The unprecedented coherent flux provided by ESRF-EBS has the potential to enable SBI at much higher X-ray energies, extending its applications to larger samples and new areas such as materials testing and palaeontology. Moreover, SBI could in the future be combined with methods like near-field ptychography or holography for higher resolution.

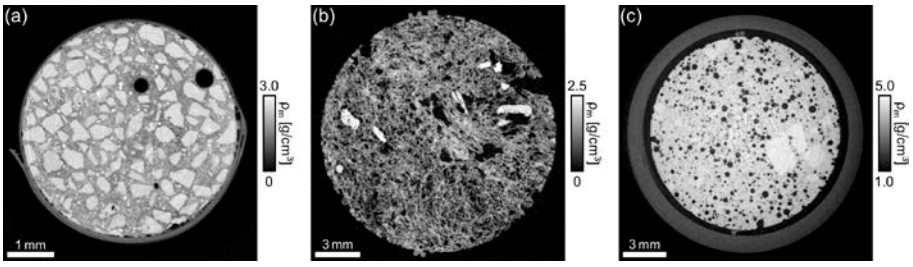

Figure 1: High-energy X-ray speckle-based phase tomography (53keV). Slices through the phase volumes of (a) a mortar sample, (b,c) different types of volcanic rocks imaged with UMPA SBI at Diamond I12 beamline.

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Holographic X-ray nano-tomography to uncover neural circuits

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Elucidating the architecture of brain neural circuits is crucial for understanding how neurons process sensorial information to generate behaviour. This is key knowledge for finding ways to cope with neurological diseases, but also for developing artificial intelligence and the next generation computing chips. Today only electron microscopy (EM) can resolve neural circuits. But to image just a fruit fly brain with EM [1] takes years of tedious work and the costs are prohibitive. Thus, access to circuits spanning over large volumes in mammalian brain remains out of reach.

We demonstrate that holographic nano-tomography (XNH) with a highly brilliant, focused and coherent X-ray probe can achieve outstanding resolving power in terms of spatial resolution and contrast in mm sized brain tissue samples [2]. This enables rapid reconstruction of dense neuronal morphologies opening new research avenues in neurosciences (Fig. 1). The coherent X-rays produced by EBS combined with cutting edge detectors and improved phase retrieval methods might enable resolving neural circuits at synaptic level which would mark a revolution in neurosciences.

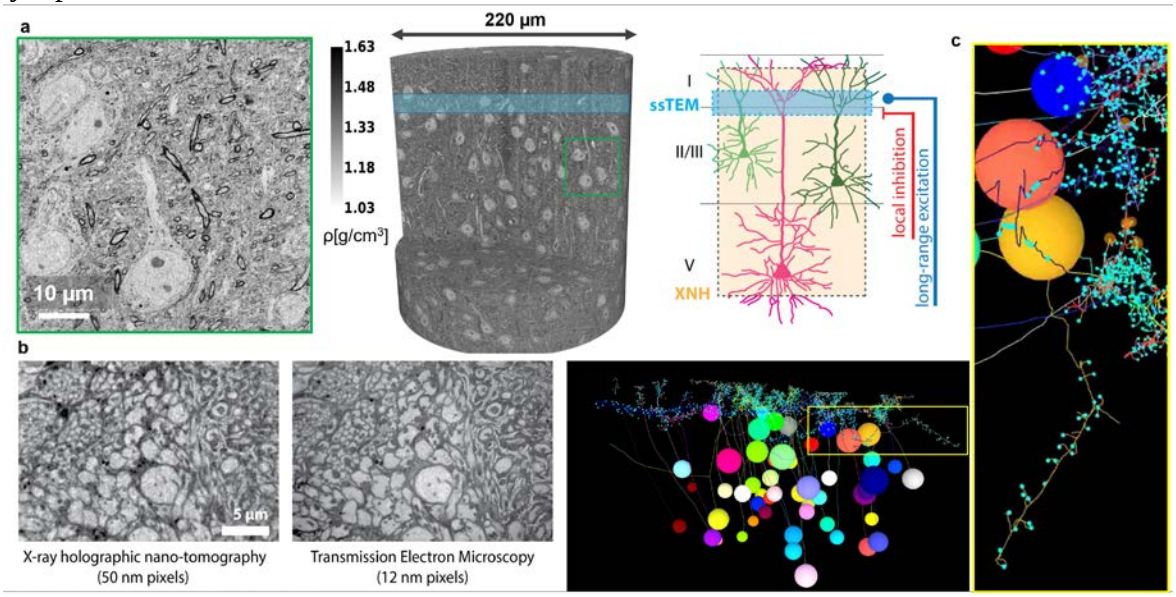

Figure 1: **a.** Tomographic slice from a mouse cortex sample imaged with voxel size of 30 nm. Dendrites, axons and cell organelles can be resolved. **b.** A *Drosophila* tissue sample imaged with both X-ray holography and electron microscopy illustrates the image quality achieved with XNH. **c.** Correlative XNH and TEM enables to discover how long-range dendrites make local synaptic patterns in mouse cortex, giving insight into how brain cortex processes information. The cylindrical volume rendering shows the volume imaged with XNH at 100 nm voxel size. The tissue region imaged with TEM after X-ray imaging is highlighted in light blue. Traced cells, dendrites and synapses are rendered in 3D using Catmaid.

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Bragg coherent diffractive imaging: an everyday microscopy tool?

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Bragg Coherent Diffraction Imaging (BCDI) is a coherent X-ray diffraction technique that allows the non-destructive probing of morphology and lattice distortion of crystalline materials with sub 10 nm 3D spatial resolution. Because it does not require optics close to the sample, BCDI is perfectly suited for insitu experiments. For example, it has been used to study phase transformations, the effects of crystal defects, crystal growth and degradation. The increase in coherent flux afforded by the ESRF upgrade will open up the opportunity to use coherent diffraction on many more instruments. As such BCDI could/should become an everyday tool for the broad, non-specialist material science community. For this to happen a number of key challenges need to be addressed:

BCDI requires that samples are crystallographically-isolated, and fit within the coherent volume of the X-ray beam (less than 1 um^3). Thus far this has limited BCDI to materials that naturally form micro-crystals of a suitable size, placing the vast majority of technologically interesting materials out of reach. Using focussed ion beam milling (FIB), we recently demonstrated the ability to manufacture micron-sized samples for BCDI from larger crystals. The FIB technique is highly site specific, making it possible to position specific micro-structural features of interest within these samples [1].

BCDI on a single crystal reflection provides one component of the lattice displacement field. By measuring at least three-independent reflections, the full lattice displacement and hence lattice strain tensor can be recovered. This is a unique capability. For reliable multi-reflection BCDI (MBCDI) measurements, the ability to quickly locate and rapidly orient specific crystals is key. We have developed approaches to this end, e.g. using micro-beam Laue diffraction to pre-orient specific crystals [2].

A major bottleneck for BCDI is reliable phasing of the collected diffraction patterns. Furthermore, the analysis of MBCDI data is complicated for samples containing crystal defects, such as dislocations, that give rise to phase jumps. Recently we have developed new tools to allow the straightforward reconstruction of the full lattice strain tensor using MBCDI, even in the presence of extended dislocation structures [1].

I will summarise recent developments in these areas, illustrated with examples and applications, concentrating on crystal defects and irradiation damage [3, 4]. I will also provide an outlook of the most pressing challenges, from a user perspective, for making (M)BCDI a general microscopy tool. These include the need for integrated sample manufacture and measurement, the urgent requirement for more robust phasing approaches and optimisation of high-throughput measurements.

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Operando Bragg CDI investigation of temperature hysteresis in CO oxidation on model gold catalysts

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Understanding catalysts dynamic behaviours is crucial for the development of cost-effective, stable and long-lasting catalysts. A peculiarity of CO oxidation reaction is the temperature hysteresis observed during a complete thermal cycle when the catalytic properties do not match during heating and cooling. Explanations about the origin of this hysteresis loop are still conflicting. In order to investigate the dynamics involved in the active sites formation and the hysteresis behaviour, we imaged gold nanocubes (~65 nm) during CO oxidation reaction by *operando* Bragg CDI at the 34-ID-C beamline at the Argonne Photon Source. The strain dynamics during the reaction as function of temperature are shown in Figure 1. It is well-known, that the lattice strain can alter the reactivity of metal surfaces: strained metal surfaces have different chemical properties from those of unstrained surfaces due the shift in the d-band center.

I will show how the anisotropic strain, occurring during the oxidation reaction, provides new understanding of the formation of the active sites in gold nanocrystals [1]. Moreover, we observed that the strain hysteresis behaviour correlates with the hysteresis loop observed during the CO_2 production. The tensile strain generation and release during the reaction directly influences the temperature hysteresis in CO oxidation.

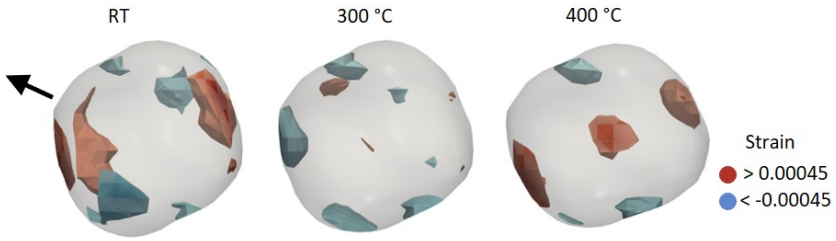

Figure 1: 3D strain field dynamics during the CO oxidation reaction, the particle shape is shown as a semitransparent gray isosurface. The red and blue regions indicate the maximum of compressive (strain < -0.0004) and tensile (strain > 0.0004) strains. The black vector is the Q_{111} scattering vector.

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In situ and *operando* structural evolution of single metallic nanoparticle model catalysts under ambient pressure reaction conditions

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Characterising the structural properties (strain gradients, chemical composition, crystal orientation and defects) inside nanostructures is a grand challenge in materials science. Bragg coherent diffraction imaging (Bragg CDI) can be utilised to address this challenge for crystalline nanostructures. A resolution of the structural properties of less than 10 nm is achieved up-to-date [1]. The capabilities of the Bragg CDI technique will be demonstrated on single nanoparticles for enhanced catalysis.

As an example, the Bragg CDI technique allows understanding the interplay between shape, size, strain, faceting [2], composition and defects at the nanoscale. We will demonstrate that Bragg CDI on a single particle model catalyst makes it possible to map its local strain/defect field and directly image strain build-up close to the facets. We will also show results obtained during *in situ* [3,4] and *operando* Bragg CDI measurements during CO oxidation and H_2 hydrogenation: it was possible to track a *single* particle in liquid and gas phase environments (see Fig. 1), to monitor its facet changes and to measure its strain response to gas or electrochemical reaction.

This technique opens pathways to determine and control the internal structure of nanoparticles to tune and optimise them during catalytic and other chemical reactions. This technique should benefit from a unique opportunity: the ESRF **EBS** Upgrade. This should revolutionise imaging by making it possible to map evolving physico-chemical processes in a slow-motion movie.

Financial support to this work by ANR Charline (ANR-16-CE07-0028-01), ANR TERC (ANR-18-ERC1-0010-01) and a NOW TOP grant is gratefully acknowledged.

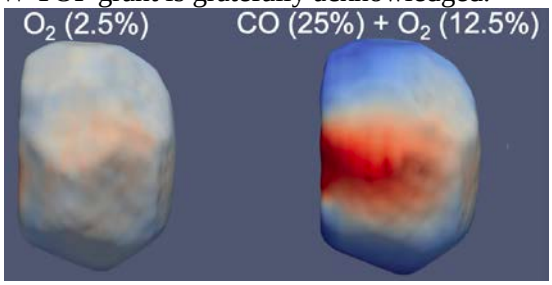

Figure 1: 3D Bragg coherent diffraction imaging reconstruction of both the shape and displacement field along the [111] direction of a facetted Pt nanocrystal (diameter of 150 nm) at 450°C during different gas mixtures: (left) in 2.5% of O_2 and (right) under stoichiometric conditions: in 25% of CO and 12.5% of O_2.

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X-ray photon correlation spectroscopy

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With its ultra-low emittance and dramatic increase in the degree of coherence ESRF-EBS will provide unprecedented experimental possibilities for coherence based applications. We will review the implications for correlation techniques such as X-ray Photon Correlation Spectroscopy (XPCS) and X-ray Cross-Correlation Analysis (XCCA) and discuss the impact of ESRF-EBS for our capabilities of characterizing the dynamical and structural properties of materials.

Complex dynamics of nanoparticles in highly concentrated entangled networks revealed by XPCS

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Nanoparticles embedded in concentrated networks exhibit complex dynamical properties determined by the topology of the network and the dynamics of neighbouring entanglement strands. Studying the processes that drive the motion of particles allows understanding and tuning the transport properties of particles through entangled or crosslinked networks and porous media. Due to the inherent complexity of those networks, the dynamics of nanoparticles is strongly length scale dependent and spans a large range of time scales. For example, aqueous mixtures of the cationic surfactant CTAB [1,2] and photoresponsive OMCA [3,4] form an entangled network of wormlike micelles [5,6] yielding a photorheological liquid. The structural and dynamical properties of these complex fluids at room temperature can be altered by illumination with UV-radiation or changing the micelle concentration [5], which allows studying nanoparticle dynamics under different confinement conditions.

We show how the complex dynamical landscape of nanoparticles in OMCA-CTAB compositions can be explored from microseconds to hundreds of seconds by means of X-ray photon correlation spectroscopy (XPCS). Results from experiments conducted at ESRF (ID10 and ID02) and PETRAIII (P10) show that the sample system exhibits complex dynamical properties depending on micelle concentration and UV-illumination time. Furthermore, we present special experimental and analysis schemes that can be successfully applied to measure extremely radiation sensitive samples, as is often the case for biological samples and soft matter.

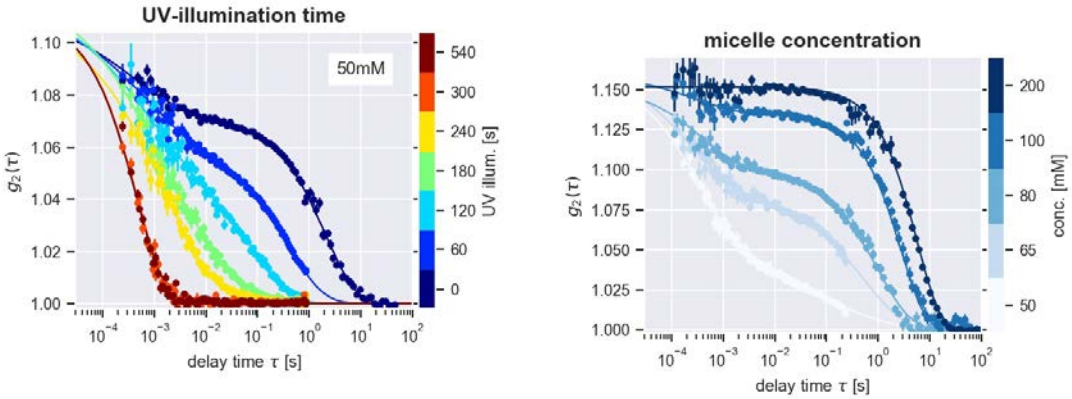

Figure 1: Correlation functions of 100nm SiO_2 tracer particles in OMCA-CTAB compositions of different UV-illumination times (left, q=0.028nm^{-1}) and micelle concentrations (right, q=0.041nm^{-1}).

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Large beam X-ray photon correlation spectroscopy

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The Coherence Beamline P10 at PETRA III is dedicated to coherent X-ray scattering experiments using X-ray Photon Correlation Spectroscopy (XPCS) and Coherent Diffraction Imaging (CDI) techniques.

Mostly, the beamline operates in the energy range of 5-17 keV. It consists of two 12 m long experimental hutches (EH1 & EH2) which house various experimental setups. Here, we present the large beam XPCS setup at P10. In this configuration, the sample position is situated in the first experimental hutch (EH1), while the detector is positioned at the end of the second hutch (EH2), which results in a sample to detector distance of around 21.3 m. This long pathway allows it to use a large fraction of the horizontal coherent flux in an unfocused X-ray beam, while providing a fairly strong speckle visibility at 8 keV using a pixel size in the range of $(75\ \mu m)^2$. The setup is therefore ideally suited for radiation sensitive samples such as most complex liquids, as the flux per sample area can be considerably reduced using a comparatively large X-ray beam.

At a photon energy of 8 keV, the minimum accessible q is around $2x10^{-4}$ $Å^{-1}$. To illustrate the possibilities for XPCS in this configuration, recent experimental results will be displayed.

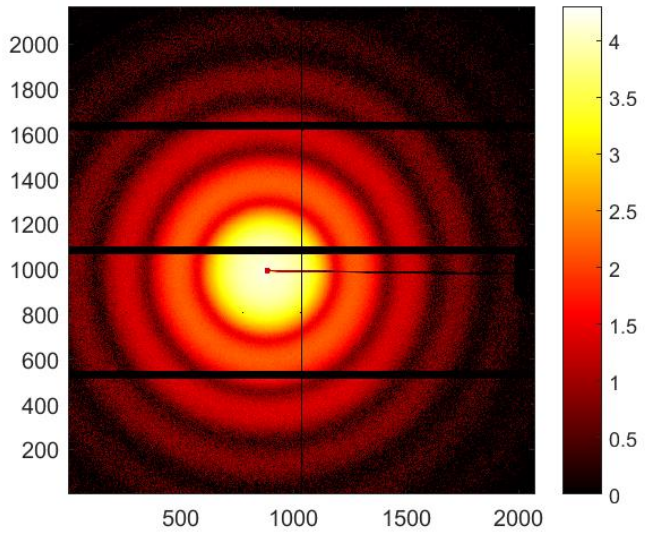

Figure 1: Scattering pattern of a nanoparticle suspension with a mean particle radius of 100 nm as detected by an Eiger X4M detector with a 155x162 mm^2 sensitive area.

Current progress and challenges in the imaging and control of Van der Waals high temperature superconducting heterostructures

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The recently newly developed techniques that allow the integration of atomically thin high temperature superconductors into Van der Waals heterostructures [1] require advanced imaging techniques, and opens the possibility of a new generation of extremely air-sensitive nanodevices for the search and study of tunable and superconductors with higher transition temperatures and their interplay with other quantum materials. Hence, advanced imaging tools and fabrication tricks are required in order to localize the sample and overcome the limitation of the ultra-thin thickness. Here we report nano X-ray diffraction imaging of incommensurate lattice structure in a bulk and high temperature superconducting Bi2Sr2CaCu2O8+y (Tc = 90 K) down to a 2 unit cells (u.c) Van der Waals heterostructure. Despite the atomically thin heterostructure (~6 nm), the superconducting critical transition temperature is identical to the bulk devices and the long- and short orders of the incommensurate dislocations of the lattice modulations are still detected. We correlate the structural and transport data by measuring the Hall-effect down to 2 u.c., establishing a quantitative agreement between theory and data. At the end of the talk, I will show our current advance in the X-ray Photon Correlation Spectroscopy (XPCS) and imaging of high temperature superconductors.

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Temporal correlations in liquid water using X-rays: From seconds to femtoseconds

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It has been postulated that the observed high- and low-density amorphous ice forms of water, HDA and LDA, can be related to two hypothesized high- and low-density liquids, HDL and LDL, through two distinct glass transitions in the ultraviscous regime. Here, we study experimentally the structure and dynamics of HDA ice as it is heated up and relaxes into the low-density form. Is this a transition between two amorphous ice forms, i.e., an HDA-to-LDA transition, or is this transition occurring between two liquid states, inferring instead an HDL-to-LDL transition in the ultraviscous regime? The unique aspect of this work is the combination of two X-ray methods, where Wide-Angle X-ray Scattering (WAXS) provides the evidence for the structural changes at the atomic level and X-ray Photon Correlation Spectroscopy (XPCS) in the small-angle X-ray scattering (SAXS) geometry provides insight about the motion at the nanoscale, respectively [1]. The diffusive character of both the high- and low-density forms is discussed among different interpretations and the results are most consistent with the hypothesis of a liquid–liquid transition in the ultraviscous regime. In addition, we will discuss of future outlook and possibilities of extending such measurements in obtaining the dynamics of liquids from seconds to femtoseconds [2] by combining Diffraction Limited Storage Rings (DLSR) with X-ray Free-electrons Lasers (XFELs).

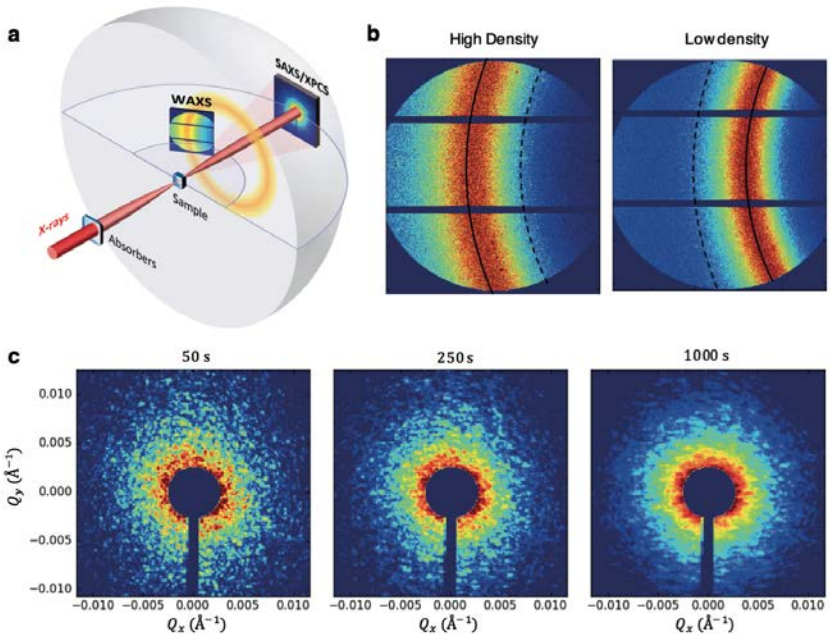

Figure 1: (a) The experimental setup combining (b) WAXS with (c) XPCS in SAXS.

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Dynamics during an arrested protein phase separation studied by XPCS and outlook for protein-XPCS at ESRF/EBS

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Dynamics in concentrated protein systems is of fundamental interest in fields such as protein crystallization, phase separation, glass transition or diffusion in crowded environments to name just a few. These systems display relatively slow and heterogeneous dynamics ranging from micro-seconds to seconds which needs to be measured on length scales ranging from micrometers down to the single-particle level on nanometer length scales. X-ray photon correlation spectroscopy (XPCS) is well suited to cover this length scale and time window employing coherent X-ray beams and tracing fluctuations in X-ray speckle patterns. We demonstrate here, that XPCS experiments of protein solutions can be performed employing X-ray doses below the damage threshold of a few kGy. With this we measured the dynamics of the antibody protein IgG in the process of liquid-liquid phase separation. Depending on quench temperature into the spinodal region of the phase diagram we observed peculiar heterogeneous dynamics displaying strong non-linear aging phenomena, increasing non-ergodicity parameters and a strong time dependence of the dynamic structure factor upon approaching the arrested phase. The dynamics mimics part of the evolution of the static structure factor but shows also distinct difference. The results demonstrate the rich information and details insights into dynamics that can be harvested from protein XPCS experiments.

Finally, we show how the superior coherence properties of the ESRF EBS coherence beamline will push the spatio-temporal window of protein XPCS experiments towards the hydrodynamic radius of a single protein [1]. Our results yield detailed information about the optimized design for future XPCS beamlines at DLSR storage rings.

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Heterogeneous and anisotropic dynamics during gel formation and aging of attractive colloidal suspensions

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Understanding the nanoscale dynamic and structural transformations during a phase transition from a freely flowing fluid to a gelled state in disordered soft materials is a fundamental physics problem and crucial to numerous technologies in pharmaceuticals, cosmetics, food processing etc. To this end, we use X-ray Photon Correlation Spectroscopy (XPCS) to investigate the microscopic dynamics during gel formation and aging in colloidal suspensions consisting of polymer-coated gold nanoparticles quenched close to and below the gelation temperature [1]. As the gel forms, a fast relaxation process (< 1s) emergent from the localized motions of the particles reveals a stiffening network on the nanoscale. The second, slower relaxation process, reflecting the gel-network dynamics, is dominated by hyper-diffusive motion of the internal stress relaxation and is well described by a compressed exponential with characteristic relaxation times inversely proportional to the wave vector transfer, q. The transient dynamics, shortly after the temperature quench, is quantified by higher-order correlations and evidence the presence of large-scale correlated motion. Temporal anisotropy develops in the network dynamics at wait times, $t_w \approx 600$ s, which subsequently vanishes. At the gelation temperature, direction-dependent dynamics indicate dynamic heterogeneity among the colloids in which unequal fraction of the particles exhibit localized motion with different amplitudes in the vertical and horizontal directions. The dynamics at temperatures below the gelation point reveal no such anisotropic behaviour, demonstrating a high temperature sensitivity of the gel-forming process.

With the opportunities enabled by the ESRF-EBS upgrade, our work motivates further XPCS experiments that would greatly advance our understanding of the complex and intricate dynamics during gelation, and in general, the formation of soft solids which are at the core of several new material technologies. For instance, one could capture the entire- two-step or multi-step -relaxation process displayed by nanoscale colloidal suspensions in a single XPCS measurement series, revealing particle motion and the gel-network dynamics over relevant length scales at time scales ranging from microseconds to a few hours [2].

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Role of higher-order spatial correlation during colloidal self-assembly

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Accessing structure beyond pair correlation functions is expected to shed light on various open questions in condensed matter physics such as the glass transition phenomenon and self-assembly processes. These phenomena are governed by the appearance of dynamical heterogeneities, suggested to be closely connected to spatial heterogeneities and believed to play a key role in the glass transition process. In order to measure such structural heterogeneities in scattering experiments, higher-order correlation functions have to be defined using e.g. the X-ray Cross Correlation Analysis (XCCA) technique.

In this contribution, we will first present results on the structure of assembled films of gold nanoparticles coated with a soft poly(ethylene glycol)-based shell. Depending on the initial concentration of gold nanoparticles, structurally heterogeneous films are formed with dominating four- and six-folds symmetry [1]. The amplitude of order parameters indicates that a minimum sample amount in necessary to obtain well-ordered structures. Furthermore, our results suggest complex symmetry-selective order formation upon ligand exchange and salt addition [2]. This richness of information cannot be achieved by standard microscopy techniques that are commonly used to characterize such nanoscale systems.

Second, we will review our recent XCCA results on the in-situ self-assembly of PbS nanocrystal superstructuress studied in real time at ID02 [3,4]. We observed the formation of hcp superlattices from the bulk suspension combining controlled solvent evaporation from the bulk solution and in situ small-angle X-ray scattering (SAXS) in transmission geometry. The application of XCCA on Bragg reflections allowed us to access information on precursor structures in the assembly process, which is not evident from conventional SAXS analysis.

Third, we will discuss the impact of the next-generation storage rings such as the ESRF-EBS on performing XCCA experiments on soft matter systems. The increase of coherent flux will enable us to reveal self-assembly processes of soft matter and nanocrystals covering all relevant length and time scales – from single particles diffusion on sub-µs over nucleation to film and crystal growth.

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Strategies to push the time resolution of X-ray photon correlation spectroscopy experiments with pixel-array detectors beyond one microsecond

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We have recently reduced the minimum time scale accessible via area detector X-ray Photon Correlation Spectroscopy (XPCS) from 1 ms to a few microseconds using a variety of increasingly more advanced area detectors and novel area detector techniques. One of these techniques exploited a Pixel-Array Detector (PAD) designed for timing experiments and capable of acquiring two gated-frames separated by a variable time delay to measure correlation functions much faster than the overall frame rate of the area detector [1]. Using another PAD, we demonstrated dead-time free continuous frame rate XPCS at 12 kHz [2] and more recently with upgraded electronics up to 52 kHz [3]. Through the flexibility of this PAD architecture, we've also demonstrated the use of a burst mode detection that achieved a peak frame rate above one MHz [4]. This presentation will review past milestones and current development with this detector, as well as recent tests of a larger version of this detector developed by Rigaku Inc.

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New opportunities to unveil glass dynamics with X-ray photo correlation spectroscopy at ESRF-EBS

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One of the most challenging issues in solid-state physics concerns the understanding of the process of dynamical arrest in soft materials and in molecular liquids. X-ray photo correlation spectroscopy (XPCS) is the only one technique able to probe the dynamics at the atomic length scale and over the typical time scales corresponding to the evolution towards an arrested state.

The XPCS technique in wide-angle configuration has been applied in recent years to the study of a variety of glass-formers, including oxide and metallic glasses [1-4]. We have now ample evidence that in the oxide glasses at room temperature the dynamics is simultaneously "pumped" and "probed" by the incident X-ray beam, with a linear relationship between the incident X-ray flux and the characteristic timescale of the induced dynamics [1,2]. In the case of the boron oxide glass, B_2O_3, we succeeded to show that it is possible to access the intrinsic dynamics of the system by increasing the temperature towards the glass transition [1]. In this way, the sample dynamics gets faster than that induced by the beam and thus experimentally accessible. However, the temporal window where the intrinsic dynamic is experimentally accessible is limited by the low coherent flux available nowadays.

In this contribution, I will review the main results we obtained in metallic glasses [4], where the dynamics shows pronounced ageing effects, and in various oxide glasses [1-3], where the effect of the beam is to induce an almost stationary dynamics.
I will then highlight the opportunities offered by the new ESRF-EBS source for XPCS on glasses. I will focus on the possibility to probe the atomic dynamics close to the glass transition and in complex sample environments, such as multi-anvil cells for high pressure studies.

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X-ray refractive parabolic axicon for coherence-related techniques: Imaging, diffraction and scattering

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The continuous development of X-ray sources, such as 3rd generation synchrotrons and X-ray free electron lasers (XFEL), has resulted in a significant increase of coherence and brightness of a photon beam, especially in the hard X-ray region. The availability of such beams led to the development of the focusing X-ray optics in order to fully utilize the beam properties, amongst which the youngest X-ray refractive optics has made a remarkable progress [1]. Today, due to the significant improvement in manufacturing techniques, the development of a new generation of X-ray optics, the optical properties of which go far beyond simple collimation and focusing functions, has become possible. This optics allows forming amplitude and phase of the wavefront with almost complete freedom, using the most outstanding properties of synchrotron and X-ray laser radiation, such as brightness, monochromaticity and coherence. This opens up new opportunities for modern X-ray studies in the field of coherent diffraction and scattering, phase-contrast microscopy and imaging, ultrafast and nonlinear optics.

For instance, we have presented the X-ray parabolic refractive axicon [2] as a clear demonstration of the beam-shaping elements operating in the high X-ray energy domain. Parabolic axicon is a biconcave lens with refractive surfaces represented in the form of a parabolic cone. Under coherent X-ray illumination, the axicon generates a Bessel-like beam propagated along the optical axis and a focused ring-shaped beam in the far field.

These optical transformations can be used in areas requiring special illumination and extended focused beams, for instance, in some metrological, medical or biological applications based on diffraction and imaging techniques. The Bessel beam is self-healing due to the conical part of the beam wavefront, meaning that the beam will reform further down the optical axis after encountering an obstruction. Based on this feature, X-ray in-line optical schemes with multiple narrow Bessel beams can be proposed, which, for example, can be used to create specific illumination in medical applications. It is worth noting that the shape and symmetry of the shaped beam image strongly depend on the astigmatism of the source. This feature was experimentally demonstrated and allows us to consider the axicon as a sensitive tool for source diagnostics and beamline alignment.

Moreover, the beam-shaping capabilities can significantly simplify some existing experimental layouts or lead to completely new optical schemes for coherent X-ray techniques based on synchrotron and XFEL sources. The annular beam generated by the axicon might substantially simplify the manipulation with beam stops in X-ray small-angle scattering experiments or in X-ray dark-field imaging techniques. Most recently, we proposed an optical scheme of phase-contrast microscopy technique based on the axicon optics [3]. Due to the unique optical properties of the parabolic refractive axicon lens, the new approach turned out to be more efficient for visualization of weakly absorbing samples as compared with the traditional microscopy technique.

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Glassy and liquid metals, from microscopic to macroscopic dynamics

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The relaxation dynamics of glass-forming metals control some of their key mechanical properties. The dynamics of the glassy state control the change from homogeneous to heterogeneous plastic deformation when applying deformation at different velocities, as well as it determines the physical aging behaviour that can produce a ductile to brittle transition. At higher temperatures, in the glass transition region and the supercooled liquid state, the dynamics control the rheological behaviour and the ability of thermoplastic forming. X-ray Photon Correlation Spectroscopy (XPCS) gives access to the microscopic dynamics of glasses form the atomic length scale up to the nanometer scale. This is the range of scales in which it is expected that the cooperative dynamics of supercooled liquids and glasses can be observed. On the other hand, mechanical spectroscopy, creep experiments and stress relaxation give access to the macroscopic dynamics [1]. An important remaining task still to do is to theoretically connect the microscopic and macroscopic dynamics, thus unveiling the atomic and nanometer-scale mechanisms of the macroscopic behaviour.

This poster presents mechanical relaxation and XPCS data obtained for various glass-forming alloys, namely $Pd_{40}Ni_{40}P_{20}$, $Pd_{40}Ni_{10}Cu_{30}P_{20}$ and $Zr_{46.75}Ti_{8.25}Cu_{7.5}Ni_{10}Be_{27.5}$, comparing the times and shapes of the macroscopic and microscopic relaxation functions in the glassy, glass transition and supercooled liquid regions, thus covering relaxation times from the order of thousands of seconds to the order of few seconds. At temperatures above the glass transition, the macroscopic dynamics are coincident in shape and time with the microscopic dynamics measured at the maximum of the structure factor. The distribution of times at this length scale is reproduced in the stress relaxation function, with similar values of stretched exponents. On the other hand, in the glassy state, the macroscopic relaxation is dependent of the level of external force applied in the experiments, changing from partial to complete relaxation at the same temperature but different levels of stress. In this case the microscopic relaxation time seems to control only the partial relaxation observed when applying low stresses. Furthermore, the broad spectrum of relaxation times observed macroscopically in this region is not reproduced in the microscopic dynamics where compressed relaxation functions are always observed.

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Holotomography for preclinical investigation in neurodegenerative diseases

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As research in neurodegenerative diseases progresses, many similarities appear that relate these diseases to one another at *cellular and molecular levels*. One of the unmet needs common to neurodegenerative diseases of the central nervous system (CNS) is the possibility to assess and monitor the fine neuropathology, like tissue inflammation and damage, throughout the disease course and upon treatment. The lack of a suitable technology able to perform this monitoring makes it therefore difficult to assess the efficacy of new therapies at the cellular level at the site of injury. X-ray phase contrast tomography (XPCT) and Holotomography allow the 3D visualization of disease-relevant networks within the CNS and to monitor inherent and infiltrating cells involved in the pathogenesis and that are of crucial importance in surveying disease progression at its earliest physiological changes, as well as in monitoring therapies.

Alzheimer's disease (AD), the most common form of dementia, is a progressive neurodegenerative disorder associated with aberrant production of beta-amyloid (Aβ) peptide depositing in brain as amyloid plaques. While animal models allow investigation of disease progression and therapeutic efficacy, technology to fully dissect the pathological mechanisms of this complex disease at cellular and vascular levels is lacking. We exploit XPCT and Holotomography to simultaneously analyse disease-relevant vascular and neuronal networks in AD mouse brain. Our findings clearly show the different typologies and internal structures of Aβ plaques, together with their interaction with patho/physiological cellular and neuro-vascular microenvironment. We detect, for the first time, amyloid-angiopathy at capillary level, which is impossible to achieve with other approaches.

The degenerative effects of **Multiple Sclerosis** at the level of the vascular and neuronal networks in the central nervous system are currently the object of intensive investigation. Preclinical studies have demonstrated the efficacy of mesenchymal stem cell (MSC) therapy in experimental autoimmune encephalomyelitis (EAE), the animal model for multiple sclerosis, but the neuropathology of specific lesions in EAE and the effects of MSC treatment are under debate. X-ray phase-contrast tomography and Holotomography enable an unprecedented direct 3D characterization of EAE lesions at micro-to-nano scales, with simultaneous imaging of the vascular and neuronal networks. We reveal EAE-mediated alterations down to the capillary network and how the disease and MSC treatment affect the tissues.

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X-ray phase contrast zoom tomography as a tool to visualize human healthy and diabetic peripheral nerves

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Conventional light and electron microscopy techniques are mostly restricted to two-dimensional visualization of tissues, such as peripheral nerves. We hypothesize that the synchrotron imaging technique obtains supplementary, and in some aspects better, morphological information and provides detailed three-dimensional (3D) images of peripheral nerves, with subcomponents, in healthy subjects and in subjects with type 1 and 2 diabetes.

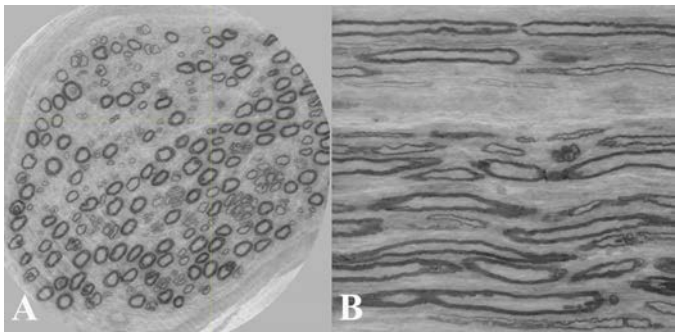

Figure 1: Subject with diabetes. Various subcomponents of the nerve, e.g. myelinated nerve fibers, myelin sheaths, axons, nodes of Ranvier, and Schmidt-Lanterman incisures can be identified. Myelinated nerve fibers in type 1 diabetes are clearly visible, simultaneously in transverse (A) and longitudinal sections (B).

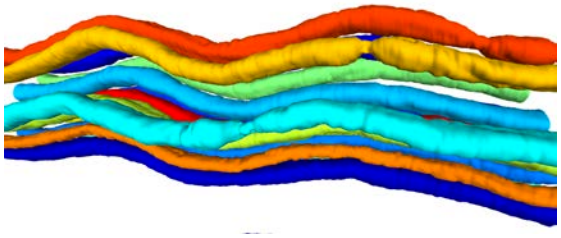

Figure 2: Visualization of segmented myelinated nerve fibers.

Biopsies of the posterior interosseous nerve at wrist levels were taken from patients with carpal tunnel syndrome in conjunction with carpal tunnel release from otherwise healthy subjects and from subjects with type 1 and 2 diabetes. Specimens were prepared for morphological analyses [1]. One biopsy from each patient category (i.e. healthy subject, subject with type 1 or type 2 diabetes) was investigated with X-ray phase contrast zoom tomography [2] at the ID16A-NI beamline of the ESRF. Excellent soft tissue contrast with an isotropic voxel size of 130 nm was obtained (Figure 1,2), revealing alterations caused by the pathology. The improved through-put, spatial and density resolution anticipated with ESRF-EBS opens the way to large scale neuropathological studies.

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Correlative Bio-Imaging at the GINIX: Scanning SAXS, Holography, and Optical Fluorescence

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We present a correlative microscopy approach for biology based on holographic X-ray imaging, X-ray scanning diffraction, and stimulated emission depletion (STED) microscopy. All modalities are combined into the same synchrotron endstation. In this way, labeled and unlabeled structures in cells are visualized in a complementary manner. We map out the fluorescently labeled actin cytoskeleton in heart tissue cells and superimpose the data with phase maps from X-ray holography. Furthermore, an array of local far-field diffraction patterns is recorded in the regime of small-angle X-ray scattering (scanning SAXS), which can be interpreted in terms of biomolecular shape and spatial correlations of all contributing scattering constituents. We find that principal directions of anisotropic diffraction patterns coincide to a certain degree with the actin fiber directions and that actin stands out in the phase maps from holographic recordings. In situ STED recordings are proposed to formulate models for diffraction data based on co-localization constraints.

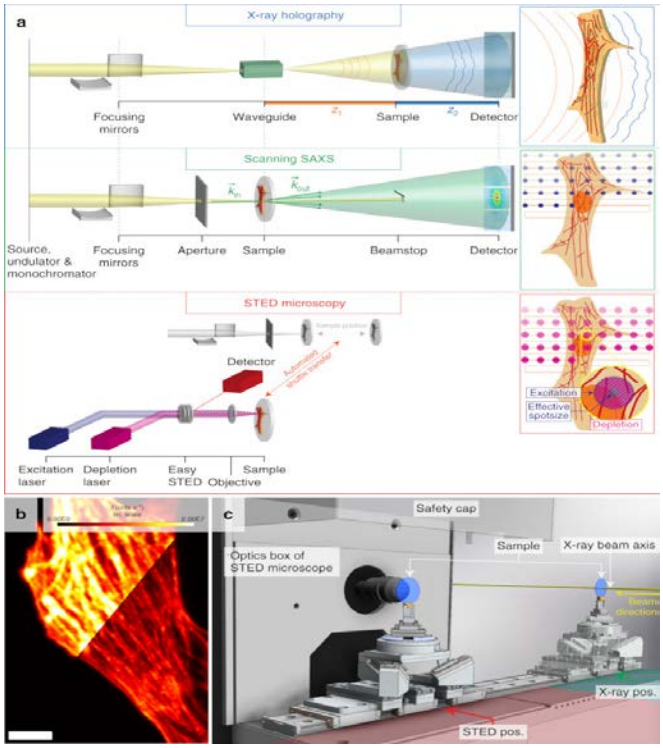

Figure 1: Recording schemes and instrumental realization at the beamline. a Schematic of the three imaging modalities: X-ray holography (top), scanning SAXS with nano-focused X-rays (center) and in situ STED microscopy (bottom). b Confocal (top left) and STED-image (lower right) of a cardiac tissue cell with labeled actin cytoskeleton. Scale bar: 5 µm. c 3D illustration of the sample in the STED (red) or the X-ray recording position (blue/green), respectively.

Successes and challenges of synchrotron X-ray nano-tomography for the characterization of solid oxide cells materials

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High-resolution characterization of Solid Oxide Cells (SOCs) materials is of central importance for a better understanding of their performances and degradation. In this frame, the complex morphology and multiple phases of the porous electrodes need to be reconstructed in three dimensions to obtain all the key microstructural properties controlling the global SOC behaviour. Coupled with long-term electro-chemical tests and numerical simulations, this enables predictive modelling of SOCs. For this purpose, synchrotron X-ray nano-holotomography appears as one of the most relevant techniques with the capability to obtain large volumes while preserving a high spatial resolution able to describe the morphological details of the microstructure. In order to adapt the holotomography method to the challenging SOC materials, a collaboration between CEA-Liten and ESRF-ID16A-beamline has been established for several years [1]. A specific sample preparation method has been developed by using a Xe Plasma Focused Ion Beam (PFIB) to control the region of extraction and the size of the analysed sample [2]. The data acquisition with random sample motion and the reconstruction algorithm have also been modified to increase the spatial resolution and to become virtually free of artefacts [3]. The improved procedure has been successfully applied to characterize the fine and coarse microstructures of the active functional and current collecting layers, respectively. The electrode microstructural evolutions upon operation have been thoroughly studied [4,5,6]. The EBS will provide new opportunities such as better contrast and higher spatial resolution required for the characterization of innovative composite electrode materials. In this frame, recent advances in the nearfield ptychography technique should allow to further improve the global quality and reliability of the reconstructions [7]. Moreover, to study the materials destabilisation and reactivity in operation, it will be highly relevant to couple the microstructural reconstructions to physico-chemical characterisations such as X-ray nano-fluorescence and nano-diffraction. Finally, it is worth noting that all the methods developed in the frame of this collaboration could be extended to batteries and more generally to materials for energy.

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Coherent diffractive imaging of non-periodic self-assembled colloidal nanocrystals

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The presentation is aimed to discuss the application of coherent diffractive imaging to probe the assembly of colloidal nanocrystals in micrometric islands, either as isolated objects [1] and as extended regions into polymers [2].

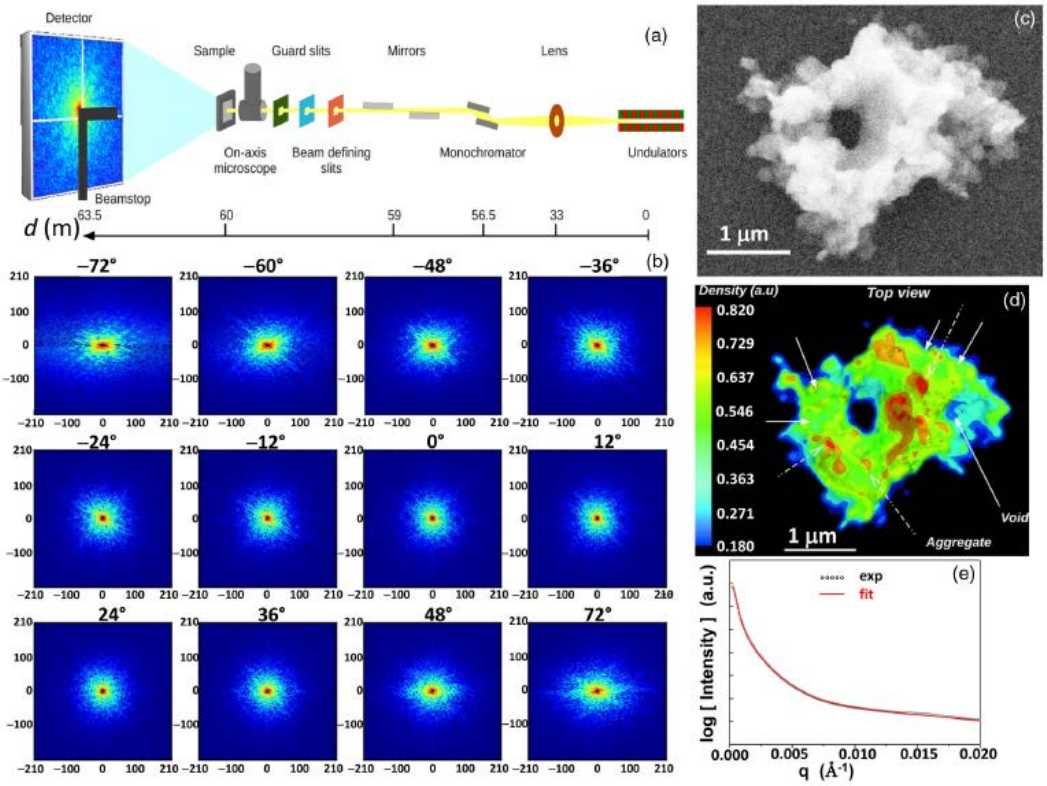

Figure 1. (a) Sketch of the experimental setup at the ID10 beam line of the ESRF reporting the distance d from the X-ray source of the different elements. (b) Example of some CDI diffraction patterns among the 73 frames taken for sample tilts between -72° and +72°with a step of 2°. (c) SEM image of the investigated Fe2P cluster. (d) Reconstructed 3D image of the sample (resolution of 59 nm). Continuous arrows show several voids; dashed arrows point to the high-density aggregates. (e) Averaged SAXS profile (experimental, black circles) and computed profile (red line) [1,3].

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Internal structure of shales by coherent X-ray diffraction imaging

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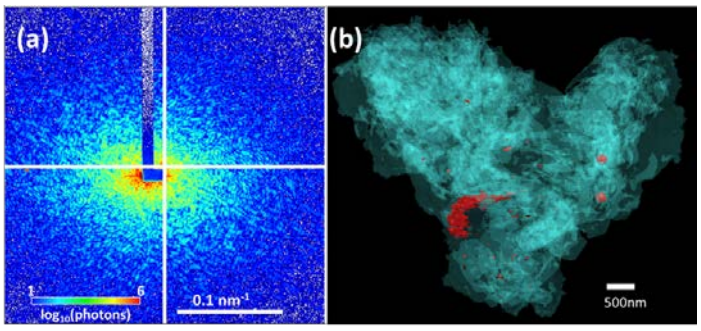
Coherent X-ray Diffraction Imaging (CXDI) is a lensless microscopy method where the real space image is reconstructed from the far-field diffraction pattern of an isolated sample provided the diffraction pattern is sampled finer than the Nyquist frequency [1,2]. Over the past decade, CXDI has emerged as an alternate 3D microscopy technique and has been successfully utilized to image diverse samples such as gold nanocrystals, biological cells, polymers etc. [1-3]. In this presentation, we describe a quantitative CXDI study of the internal structure of shale cuttings demonstrating the use of CXDI to image such specimens. As part of our long-term objective of understanding the structural heterogeneity of shales over different length scales, Pierre Shale 1 (a typical shale from South Dakota USA) [4] is studied here as a model system due to its mineralogical heterogeneity as well as the complex porous network. CXDI and wide-angle X-ray diffraction (WAXD) measurement were performed in tandem at the ID10 beam line of the ESRF, France [5]. The combined use of CXDI and WAXD enabled 3D reconstruction of the electron density distribution across the entire sample and identification of the crystalline phases. In addition, the nanoscale pore structure was studied. Given in Fig. 1 are the diffraction pattern and the reconstructed image of a shale fragment. The results allowed understanding of the 3D microstructure of shale particles and establishing structure-property correlations.



Figures: (a) Diffraction pattern collected in the far-field regime and (b) 3D iso-surface rendering of the reconstructed sample. Regions of high electron density are colored red.

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Structural characterization of TiO_2 gig-lox sponges by coherent X-ray diffraction imaging

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We created a blend between a TiO_2 sponge with bimodal porosity and a $MAPbI_3$ perovskite. The interpenetration of the two materials is effective thanks to the peculiar sponge structure. During the early stages of the growth of the TiO_2 sponge, the formation of 5-10 nm-large TiO_2 auto-seeds is observed which set the nano-porosity (< 5 nm) of the layer, maintained during further growth. In a second stage, the auto-seeds aggregate into hundred-of-nm-large meso-structures by their mutual shadowing the grazing Ti flux for local oxidation[1,2,3,4,5]. This process generates meso-pores (10-100 nm) treading across the growing layer, as accessed by tomographic Synchrotron Radiation Coherent X-ray Imaging and Environmental Ellipsometric Porosimetry. The distributions of pore size are extracted before (> 47%$_V$) and after $MAPbI_3$ loading, and after blend ageing unfolding a starting pore filling above 80% in volume. The degradation of the perovskite in the blend follows a path towards the formation of PbI_2 with the concomitant release of volatile species with an activation energy of 0.87 eV under humid air. The use of dry nitrogen as environmental condition has a positive impact in increasing this energy by ~0.1 eV that extends the half-life of the material to 7 months under continuous operation at 60°C.

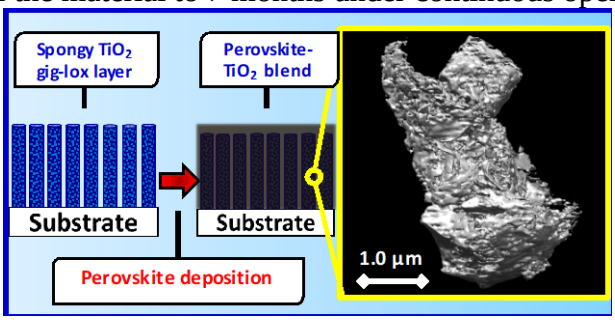

Figure 1: CXDI 3D reconstruction of a TiO_2 gig-lox portion functionalized with $MAPI_3$ perovskite.

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3D analysis of bone ultra structure from magnified phase nano-CT

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The prevalence of bone diseases such as osteoporosis requires a better understanding of bone fragility mechanisms, relying on a multi scale assessment of bone tissue. The investigation of bone tissue at the cellular and ultrastructure scale is receiving increased attention, nevertheless there are few 3D and quantitative data at this scale [1].

X-ray imaging is increasingly used together with biomechanics to assess bone strength at different scales. At the microscopic scale, synchrotron radiation (SR) micro-CT permitted a detailed analysis of bone microstructure together with bone mineralization. By further exploiting the coherence properties of the beam, SR propagation based phase CT opens interesting perspectives to quantify bone ultrastructure at the nanometer scale.

In this work, we investigated the bone lacuno-canalicular network (LCN) from magnified X-ray phase nano-CT at 120 and 30nm. Image acquisition was performed on beamline ID16A at the European Synchrotron Radiation Facility (ESRF).

First, we studied the impact of the number of propagation distances and phase retrieval algorithms on image quality. Our results showed that the best quality was achieved when using the extended Paganin's multi distance algorithm followed by an iterative non-linear refinement [2]. Then, we developed an image analysis workflow to segment and extract quantitative parameters of the LCN. While the segmentation of lacunae was relatively straightforward, the segmentation of canaliculi, corresponding to very small sizes channels required a dedicated process based on vesselness enhancement, Region Growing (VRG) and connected components filtering to clear out residual noise [3]. Quantitative parameters allowing to estimate the numbers and size of lacunae and canaliculi were measured. We present results on novel 3D characteristics of the LCN obtained on human samples from images acquired with a voxel size of 120 and 30nm (see Figure 1).

In conclusion, magnified X-ray phase nano-CT is an efficient technique to analyze bone ultrastructure and allow us to learn about the mechanisms of bone fragility in disease.

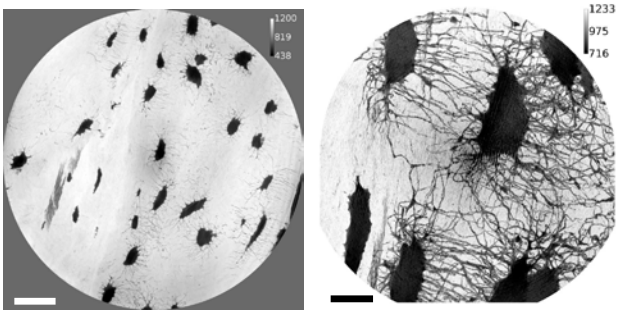

Figure 1: Illustration of a MIP display of the LCN from a phase nanoCT image at 120nm(left), 30nm (right).

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Fast *in situ* 3D nano-imaging at the ESRF

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Nano-characterization is a key point in the study of microstructures for the understanding of material properties, as well as its evolution and degradation. More and more, *in situ* measurements are needed to further understand the mechanisms controlling these processes. X-ray computed tomography (CT) is a very powerful technique that provides nondestructively direct access to the three-dimensional morphology of a specimen, and for which recent improvements have led to measurements at the nano-scale. As an example, the two beamlines, ID16A and ID16B at the European Synchrotron Radiation Facility (ESRF) take advantage of the holotomography technique [1] to 3D image materials from polymers to ceramics, at the nanometer scale (pixel size down to 10nm). In this work, we present the unique fast *in situ* 3D nano-imaging tool developed at ID16B combining high spatial resolution and time resolved measurements.

The unprecedented combination of high spatial and temporal resolution (timescan:7s; pixel size 27 nm) have been made possible thanks to the unique characteristics of the ID16B beamline:the intense, high energy, nano-focused X-ray beam (10^{12}ph/s - energy from 17 keV to 30keV - 50×50 nm^2) and its in air sample environment. Thanks to this last characteristic, it has been possible to develop new and complex sample environments, such as furnace to perform *in situ* 3D nano-imaging at high temperature, Fig. 1. A recent improvement has been the development of a tensile device that can be used into the furnace to follow temperature deformation. Several experiments have been performed on light alloys to follow creep cavities. The results show the nucleation and early stage of the growth of cavities (in red) near particles (in green) in Aluminum alloys during tensile tests at 400°C, Fig. 2. Changes in shape and volumetric data have been linked to theoretical models such as diffusion and plasticity [3].

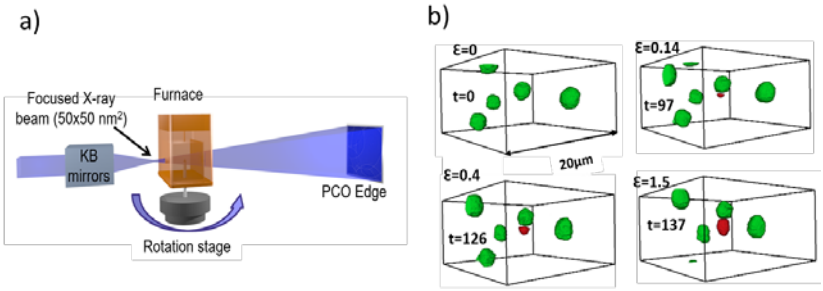

Figure 1**:** (a) Fast high temperature set up at ID16B: beam of 50x50 nm2 focused down thanks to KB mirrors, fixed PCO edge **(**b) 3D rendered view of nucleation and growth of cavity (red) alongside a second phase particle (green) as the Al-Cu sample strains (Ɛ) with time (t, in minutes), imaged by *in situ* nano-tomography with a voxel size of 100nm during tensile test at 400°C.

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Future opportunities for XPCS of atomic-scale dynamics at interfaces

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The greatly increased coherent X-ray flux available from MBA storage rings will enable X-ray photon correlation spectroscopy (XPCS) studies of a much wider variety of systems and phenomena than currently possible. These include more weakly scattering systems, such as surfaces and 2-d materials, and observations of short-length-scale dynamics at higher wavenumber and at the associated higher speeds. In addition, the increased coherent flux at higher X-ray energies will allow better optimization of the trade-off between higher signal level and undesired interaction of the X-ray beam with the sample dynamics. In this talk I will discuss some of the scientific opportunities for studies of atomic-scale dynamics at interfaces. These will be illustrated by examples of recent state-of-the-art experiments (e.g. [1]) as well as simulations of surface phenomena and XPCS.

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Liquid/liquid interface dynamics probed by grazing incidence XPCS

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Albeit the structural evolution accompanying the sol-gel transition of sodium alginate has been largely investigated [1], no studies have been reported on the corresponding dynamics of interfacial gelation at the o/w interface. Several pioneering studies suggested that grazing incidence X-ray photon correlation spectroscopy, GI-XPCS, is one of the new challenges to shed light on the dynamics of soft interfaces [2]. In this study, interfacial gelation of sodium alginate at the o/w interface and the temporal evolution of the underlying dynamics at the nanometric length scale were investigated by means of GI-XPCS. Similar to what was previously done for methylcellulose [3], in a first step, the sol-gel transition of alginate was detected in bulk (SAXS-XPCS) by looking into the dynamics of silica nanoparticles used as tracers in the polymer matrix. The results clearly indicate that the characteristic relaxation time calculated from the autocorrelation functions of the silica tracers strongly depends on the cross-linker concentrations. Subsequently, to monitor the dynamics at the interface, we utilized chemically modified SiO_2 nanoparticles confined at the o/w interface as well as the sample environments for o/w experiments available at ID10 [4]. The autocorrelation functions calculated from the scattering intensities at different times after cross-linker injection clearly indicate a significant change in the characteristic relaxation time over one order of magnitude. The obtained results served as a proof of concept with respect to GI-XPCS applied to soft liquid-liquid interfaces. Further investigations are necessary to shed light on the kinetics of interface gelation from the viewpoint of interface dynamics.

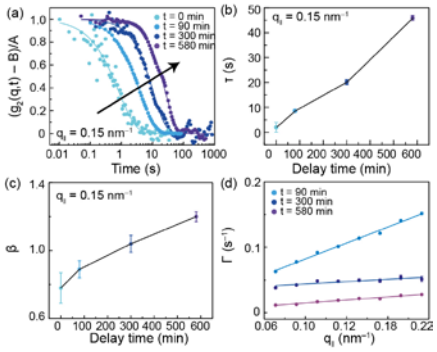

Figure 1: Change in interfacial dynamics of sodium alginate at o/w interface unraveled by GI-XPCS.

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Grid-type distorted born approximation: A dynamic scattering model for coherent surface scattering imaging

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Coherent surface scattering imaging (CSSI) is a novel hard X-ray technique that can probe 3D non-periodic structures, buried or on-surface, at very high resolution (5nm in-plane and <1nm out-of-plane). The potential will be further boosted thanks to the next generation diffraction-limited synchrotron sources such as the EBS and the APS-Upgrade. Due to the grazing-incidence reflection geometry required by CSSI, photons are more likely to be scattered multiple times, violating the Born approximation which equates the scattering intensity with the modulus squared of the sample electron density profile's Fourier transform, the essential assumption adopted by coherent diffractive imaging (CDI). Distorted Wave Born Approximation (DWBA) which is widely used to describe the dynamic scattering phenomena for thin films, fails for CSSI samples which can be highly non-uniform and anisotropic. In this talk, we'll present a grid-type DWBA model that divides the sample into a mesh grid, calculates the electrical field for each element using the normal DWBA, and sum the scattering cross-section within each element as well as the scattering between nearby elements. This method is able to generate high-fidelity scattering patterns that's similar to our experiment with much less computational effort compared to the finite difference time domain (FDTD) technique. We expect this method finds applications in understanding, predicating and even reconstructing the surface coherent scattering data from CSSI and grazing incidence diffraction experiments.

Posters

Workshop on Coherence at ESRF-EBS
9 to 13 September 2019
ESRF Grenoble

List of posters

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I. Bukreeva	Phase contrast X-ray tomography for investigation of spine and spinal cord of ex-vivo small animals
G. Chahine	Coherence on F-CRG BM beamlines. Is it possible?
P. Cloetens	Coherent X-ray imaging at ID16A: status and plans
J.C. Da Silva	X-ray spectral-ptychographic imaging for the characterization of technical catalyst bodies
D. Dzhigaev	Nano-scale strain mapping in complete nanowire based electronic devices by Bragg coherent X-ray diffraction
V. Favre-Nicolin	Online, quantitative data analysis for coherent X-ray imaging techniques with the ESRF PyNX toolkit
M. Fratini	Multimodal approach for the investigation of the CNS
B. Gerbelli	Thermal and structural characterization of hybrid hydrogels composed of amidogenic and PEG-silanized peptides
G. Girard	Combining diffraction contrast tomography and Bragg coherent diffraction imaging on embedded micron-sized ceramic grains
P. Godard	In-situ compression of InSb micro-pillars under coherent X-ray diffraction
C. Gramaccioni	Correlative 3D structural and chemical quantification of single human cells
Z. Jiang	Reconstruction of X-ray waveguide fluorescence holography for evolving nanostructures in thin films
K. Kazarian	Pulsed magnetic field setup for temperature dependent dynamics studies
S. Leake	ID01 in light of the ESRF-EBS
F. Livet	Dynamic critical scattering by XPCS in the ordering AuAgZn2 alloy
F. Meneau	Cateretê, the coherent scattering beamline at Sirius, 4th generation Brazilian synchrotron facility
J. Möller	The coherent applications instrument MID at European XFEL - commissioning and first user
M. Osterhoff	Multi-order imaging with multilayer zone plates
M. Osterhoff	Focus characterization of the NanoMax Kirkpatrick-Baez mirror system
F. Peyrin	Investigation of osteons properties in human cortical bone using X-ray phase micro-CT
E. Pineda	Glassy and liquid metals, from microscopic to macroscopic dynamics
C. Polo	Bagasse nano-structure modifications induced by hydrothermal pretreatment revealed by CXDI
J. Schwenke	SoftiMAX - A new soft X-ray microscopy and coherent imaging beamline at the MAX IV facility
A. Snigirev	A new X-ray reflecto-interferometer based on refractive optics
M. Sutton	Measuring speckle q-q correlations
M. Voevodina	X-ray thin-film interferometry technique using an X-ray microfocus laboratory source
W. Vos	Placing quantum dots in 3D photonic crystals and finding them back
W. Vos	X-ray imaging of functional three-dimensional nanostructures
F. Westermeier	Large beam X-ray photon correlation spectroscopy
D. Yang	Mapping data between sample and detector conjugated spaces in Bragg
E. Zatterin	3D Bragg CDI of a pentagonal bipyramid twinned Au crystal

Coherent imaging at MAX IV

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The MAX IV Laboratory operates the first of the diffraction-limited storage rings (DLSR:s) that are now being built or designed worldwide. The high-brilliance source is particularly well suited for coherence applications such as ptychography and coherent diffraction imaging. The hard X-ray nanoprobe NanoMAX is currently taking full advantage of the coherent beam available at the synchrotron. The soft X-ray beamline SoftiMAX will begin operations by the end of this year, and also provide new coherent imaging opportunities.

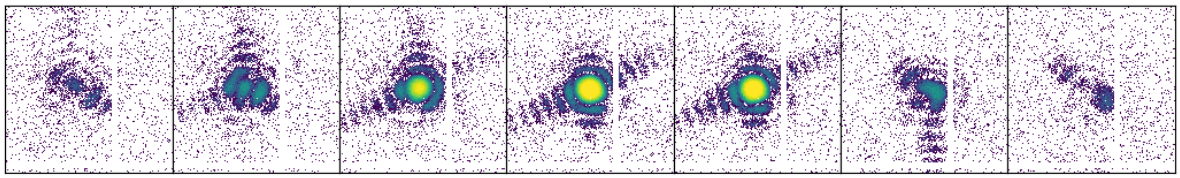

Figure 1: Coherent Bragg diffraction from a single nanoparticle encodes its 3D shape and strain state.

The first user operation campaigns at NanoMAX has seen user experiments in two main categories. First, many users have applied the coherent beam condensed matter physics problems with emphasis on semiconductors and strain characterization. Secondly, scientists from diverse parts of biology and geology have used the nanofocused beam for fluorescence mapping. Since the KB-mirror endstation provides a relatively high flexibility, users are increasingly providing sample environments for measurements under specific electrical, magnetic, thermal, pressure, or chemical conditions.

Phase contrast X-ray tomography for investigation of spine and spinal cord of ex-vivo small animals

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The modern high-resolution 3D imaging techniques such as X-ray Phase Contrast micro-Tomography provide great insight into the internal structure of objects. However, given the complexity of biomedical objects, it is often difficult for researchers to interpret and analyze data in a meaningful and efficient manner without destructive preparation of the samples. We present here Phase Contrast micro-Tomography imaging combined with relevant computational platform to get high-resolution and high-contrast 3D images of the spinal cord without the need of contrast agents, or of destructive sample preparations such as removing the vertebrae in ex-vivo small animal models. In addition, we proposed and tested Simultaneous Iterative Reconstruction method with Total Variation regularization as a tool to reduce X-ray exposure time during the scan and prevent the motion artefacts formation in high-resolution 3D image of the spinal cord. We demonstrated that micro-XPCT with sparse set of tomographic projections preserves the visualization details in spinal cord surrounded by vertebrae.

Coherence on F-CRG BM beamlines. Is it possible?

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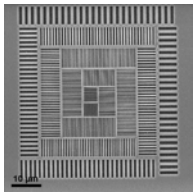
Coherent X-rays are becoming powerful tools for imaging complex, heterogeneous structures with spatial resolution below focused synchrotron beam sizes. Many beamlines are designed and optimized for exploiting the coherence of X-rays and are mainly based on insertion devices as photon source. The combination of the source size together with long source-sample distance increases the coherence length and defines the size of fully coherent beams. Due to flux limitations, bending magnet beamlines are often considered inefficient for coherent imaging. Nevertheless, the latest developments in single X-ray photon counting detectors will enable bending magnet beamlines to exploit the coherence of the X-ray photons. A test measurement performed at the French CRG D2AM (BM02) beamline showed the possibility of detecting well-resolved coherent fringes from a structured tungsten pattern in transmission geometry.

In addition, the ESRF EBS upgrade will offer 15 times smaller sources with the new SBM (short bending magnet) which will replace some of the existing BM. This reduction in source size will further increase the coherence length and thus increase the flux in a fully coherent beam.

(a)



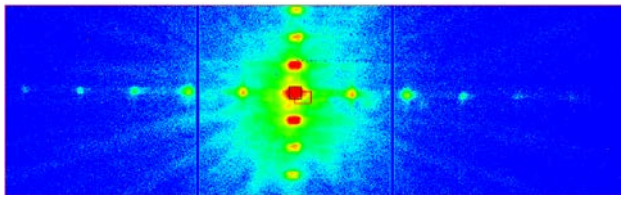


Figure 1: (a) SEM image of multiscale tungsten structures. (b) Diffraction pattern from the tungsten structures.

With the combination of high detection sensitivity and increase of coherence length, the F-CRG D2AM and FAME beamlines will offer to their community the possibility of performing coherent imaging and thus extend their capabilities in characterizing complex compounds and devices.

Coherent X-ray imaging at ID16A: status and plans

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Designed for quantitative three-dimensional characterization of the morphology and the elemental composition of specimens at the nanoscale, the ID16A-NI beamline of the ESRF produces currently the world's brightest nanofocus. With the endstation located at 185 m from the source, the beamline is optimized for coherent hard X-ray imaging and X-ray fluorescence microscopy. The instrument offers exceptional focusing down to 13 nm with a very high photon flux (up to 10^{12} photons/s at $\Delta E/E$~1%) [1]. The selected energies, 17 keV and 33.6 keV, are well suited for applications in biomedicine, materials science and nanotechnology. Two coherent imaging techniques, X-ray holographic [2-4] and ptychographic [5-6] tomography, provide the electron density distribution at length scales ranging from ~130 nm down to ~10 nm, while keeping a relatively large field of view. Complementary, X-ray fluorescence microscopy delivers label-free, highly efficient trace element quantification [7].

The instrument attains its unique properties by combining efficient nanofocusing optics (multilayer coated fixed curvature Kirkpatrick-Baez mirrors) with a carefully designed mechanical device for stable sample positioning and accurate scanning. The optics introduce strong wavefront inhomogeneities, which are handled by a specific holographic acquisition scheme [8]. Alternatively, the inhomogeneities can be exploited as a form of structured illumination in near-field ptychography [9]. The samples are measured in vacuum and the system enables correlative phase contrast – fluorescence microscopy [10-11]. All measurements can be performed under cryogenic conditions to preserve the biological samples close to their native hydrated state and reduce radiation damage. Recently, complementary cryo correlative light microscopy has become available, providing unambiguous identification of specific organelles. The cutting-edge capabilities of this instrument enable unprecedented research studies in biomedicine, materials science, and nanotechnology, thus opening new scientific frontiers.

The new ESRF source is expected to provide up to two orders of magnitude increase in brilliance and coherent fraction of the X-ray beams. This 'Extremely Brilliant Source' will be ideally suited for ultrafast, nanoscale, coherent imaging applications. In this context, we will present the current achievements and possible future directions of the beamline.

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X-ray spectral-ptychographic imaging for the characterization of technical catalyst bodies

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X-ray Spectral-ptychography is a combination of ptychography and spectroscopy and this approach is emerging in synchrotron facilities. One important domain of application is the characterization of technical catalysts. One example is the Fluid Catalytic Cracking (FCC) bodies, which are very important industrial catalysts in oil refinery. We have already characterized the morphology of FCC catalytic particles by standard ptychographic-tomography (Fig. 1) [1,2]. However, metals such as Nickel (Ni) poison the active sites of the FCC catalyst bodies during operation in the FCC reactor. This leads to problem of decreased activity and selectivity as well as increasing coking problems. Therefore, the characterization of the distribution of metals and their speciation within the particle structure can help to optimize the catalyst structure in order to reduce this poisoning and extend the time-of-life of those catalysts. The spatial resolution in classical spectral imaging is limited by the beam sizes available, which is not high enough for this characterization. Spectral-ptychography, on the other hand, enables morphological and spectral information of the samples with high-resolution, independent of the beam size used for the imaging. Here we present a demonstration of spectral-ptychography carried out at ID16B beamline of ESRF for the characterization of FCC catalyst bodies. We performed the experiment across the Ni K-edge, which is 8.333 keV, to identify the locations and speciation of Ni within the sample. In the context of the ESRF-EBS, this innovative technique opens new opportunities for the characterization of heterogeneous materials, not only catalysts, in which the correlation between morphology and spectral information is necessary.

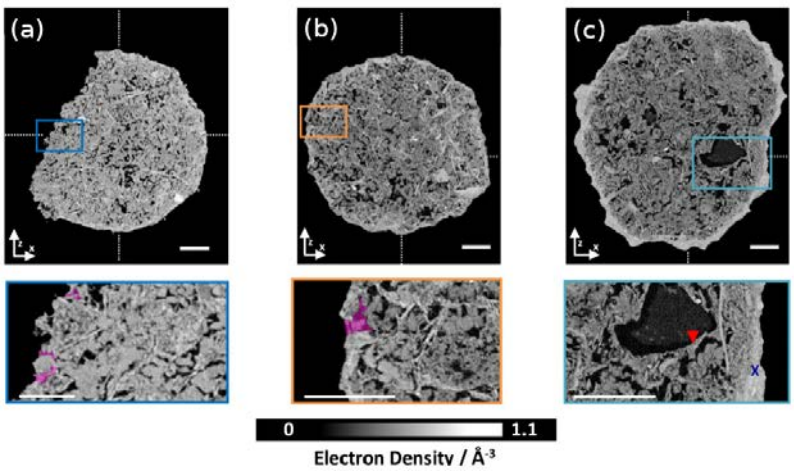

Figure 1: FCC catalyst bodies at three stage of life: (a) pristine; (b) middle life; (c) deactivated and contaminated with coke.

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Nano-scale strain mapping in complete nanowire based electronic devices by Bragg coherent X-ray diffraction

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The progress achieved in a growth of heterostructured nanowires led to design of nano-electronic devices with record-breaking parameters in transistors, quantum computations, solar cells and light emitting technologies. The strain plays significant role in the performance of these nano-electronic devices. In multi-segment nanowires the strain induced at the interfaces significantly change a charge carrier mobility, thus alternating the speed and power consumption during operation [1]. The only way to image the strain in real functional device with many functional layers and over extended field of view is to employ scanning techniques with the use of nano-focused X-ray beams. We made a deeper look at nanoscale features within single heterostructured nanowires, which are embedded in realistic nano-electronic devices. These are InAs/GaSb segments of Tunnel Field Effect Transistors (TFETs) [2] which are covered by processing layers of the metal gate, organic space and top metal contact.

Exploiting extraordinary brilliance of 4th generation synchrotron MAX IV (Lund, Sweden) and dedicated nano-focusing end-station NanoMax we could obtain the information which was not accessible before at resolution of 60 nm and below. Development of Bragg diffraction mapping and Bragg ptychography techniques in recent years opened exciting possibilities for 3D imaging of strain and tilt in extended structures [3,4]. These techniques were employed during our studies. Particularly the influence of the metal gate deposition process on the strain within a single nanowire transistor was revealed. The experimental challenges will be discussed in more detail. Interpreting of the results plays a significant role in obtaining quantitative information, which includes developing of proper models for strain engineering in nano-electronics. In our case, we support the experimental findings by Finite Element Method simulations which take into account material properties, boundary conditions and allow precise simulation of the diffraction experiment.

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Online, quantitative data analysis for coherent X-ray imaging techniques with the ESRF PyNX toolkit

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The upcoming Extremely Brilliant Source (EBS) upgrade will provide two orders of magnitude more coherent photons, and will give the ability to collect coherent X-ray imaging datasets faster and/or with a higher resolution. Consequently, the increased volume of data requires dedicated tools to fully take advantage of the improved coherent flux.

In this presentation we will discuss the PyNX toolkit[1], which is developed at ESRF to provide fast (GPU-accelerated) and accessible (using simple command-line scripts or notebook) data analysis for a wide range of experimental techniques:

- Coherent Diffraction Imaging (CDI) and Ptychography (far field and near field) for two and three-dimensional imaging
- 3D CDI and Ptychography in the Bragg geometry to also provide strain information in nano-crystals
- Phase contrast imaging

We will present examples of analysis on various types of samples (crystalline or not), and show how the available tools aim to remove the need to master coherent X-ray imaging techniques to exploit them. This will ultimately enable a wider community to take advantage of the increased coherent photon flux, and focus on working on an extended range of applications and samples.

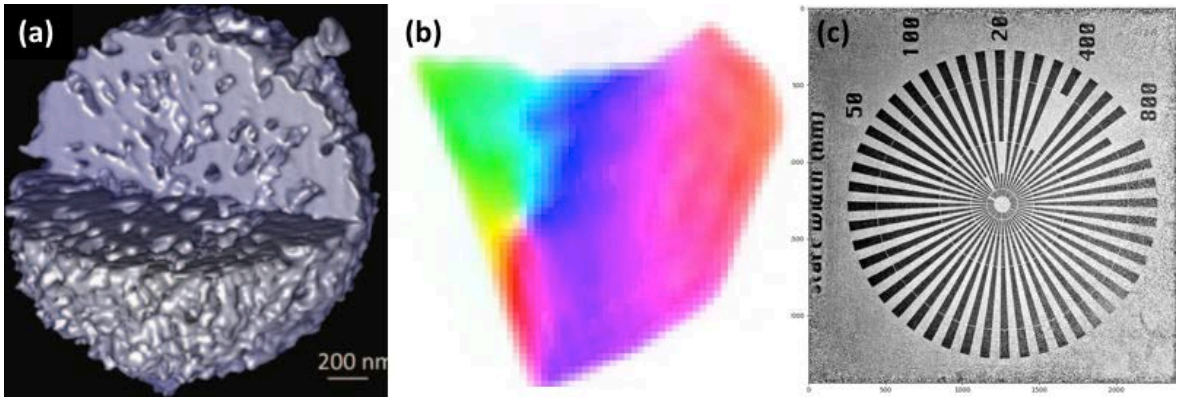

Figure 1: Examples of data which can be analysed using PyNX: (a) Coherent diffraction imaging of a calcium carbonate sample [2] (b) Bragg coherent diffraction imaging of a Pt nano-crystal with a dislocation and[3] (c) near-field ptychography imaging of a Siemens star standard target [4]

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Multimodal approach for the investigation of the CNS

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The spread of neurodegenerative diseases, such as Alzheimer's disease, Parkinson's disease, amyotrophic lateral sclerosis, multiple sclerosis and Huntington's disease, represents one of the most prominent health problems worldwide. Recently, there was a growing interest in high-resolution imaging techniques for the identification of several pathological markers, signalling the presence of neurodegenerative diseases.

Within this framework, we developed a multidisciplinary network between X-ray advanced imaging techniques, functional magnetic resonance imaging and new algorithms to develop a solid multimodal method for pre-clinical research. This will allow for a direct, quantitative estimation of important morphological and topological parameters characterizing the vascular and neuronal networks in the spinal cord (SC). In particular, the 3D SC geometry obtained using SXrPCuT, in combination with specifically designed algorithms, will tackle the study of the SC vascular and neuronal networks, which together contribute to the blood oxygenation level dependent (BOLD) signal in functional Magnetic Resonance Imaging (fMRI). This will greatly aid the regular application of SC fMRI in the clinical practice, e.g., for the early identification of spinal cord injuries and neurodegenerative diseases.

This work was founded by the Italian Ministry of Health under the Young Researcher Grant 2013 (GR-2013-02358177)

Thermal and structural characterization of hybrid hydrogels composed of amidogenics peptides and PEG-silanized

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Peptide hybrid hydrogels are a promising alternative for obtaining nanoscopic biocompatible structures. The variety of combinations between different amino acids allows the construction of a vast number of supramolecular architectures with equally diverse physicochemical properties. Thus, combining hybrid copolymers with biomolecules makes them interesting for biomedical applications such as tissue engineering and drug carrier systems. In this work a new class of hybrid hydrogels was obtained by the sol-gel process, based on an amyloidogenic peptide sequence composed exclusively of arginine, phenylalanine and glycine (Gly [Arg-Phe]$_4$).

This was synthesized and functionalized with an alkoxysilane via phase solid, then covalently bonded to the silanized polyethylene glycol. The thermal tests allowed us to observe that the presence of the peptide in the hydrogel increases the number of cross linkers and a glass transition has emerged that indicates increased mobility between the PEG polymer chains. Rheological properties showed that hydrogels have predominantly elastic characteristics, with storage modulus (G') greater than loss modulus (G”) in all compositions and demonstrated thixotropic property for peptide conjugate samples.
We also performed experiments small angle X-ray scattering (SAXS) which brought information about structures in the nanometer range, we propose a model fit the experimental curves showing that the system has two independents from each other morphologies. The images by electron microscopy and atomic force images revealed amorphous and fibrillar regimes depending on the concentration of peptide-silane in the hydrogel matrix corroborating with the SAXS results.

Acknowledgment
This work was supported by FAPESP (grant nos. 2015/24018-1, 2014/50972-1 and 2017/02317-2) and CNPq (grant no. 302923/2015-2). INCT in Bioanalytics (FAPESP grant no. 2014/50867-3 and CNPq grant no. 465389/2014-7) is gratefully acknowledged for the grants. B.B.G. acknowledges FAPESP (project number 2018/05888-3) for a postdoctoral fellowship.

Combining diffraction contrast tomography and Bragg coherent diffraction imaging on embedded micron-sized ceramic grains

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Residual stresses and strains have a large impact on the mechanical properties of ceramic materials,affecting their ultimate strength and fatigue properties. While the macro-and microscopic stress/strain relationship has long been studied, the ability to study strain fields at the grain level has so far been limited. This poster presents a study of a 20 µm diameter cylinder of Cerium doped Zirconia containing grains of a few microns in size.

Based on the Bragg Coherent Diffraction Imaging (BCDI) techniques [1], we studied the feasibility of retrieving the 3D shape and strain tensor of micrometer-sized grain in the bulk of a polycrystalline specimen after indexing by means of Diffraction Contrast Tomography (DCT) [2]. At the ESRF beamline ID01, the grain mapping from DCT allowed us finding quickly a specific grain in its diffraction condition (Fig. 1), from which we collected full rocking curves (0.025° step, 1.2° range) to be reconstructed with BCDI algorithms (Fig. 2).

While reconstruction of nanocrystalline objects using Bragg CDI has been successful on a subset of samples like isolated nanocrystals/wires and epilayers and is becoming more accessible with the development of more user-friendly algorithms and software for data analysis [3], the technique remains challenging for other types of samples, e.g grains of micrometer size matching the one of the defocused beam (> 1um). In these cases, the longitudinal coherence length of the X-ray beam becomes the essential limitation, and we will discuss to what extent quantitative information can be retrieved.

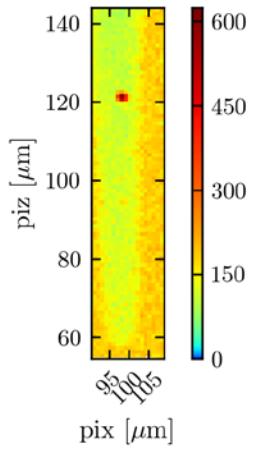

Fig.1. Fast k-map [4] of piezo stage to find a grain along a ZrO2 cylinder (integrated intensity near a 111 reflection). (Coordinates in um)

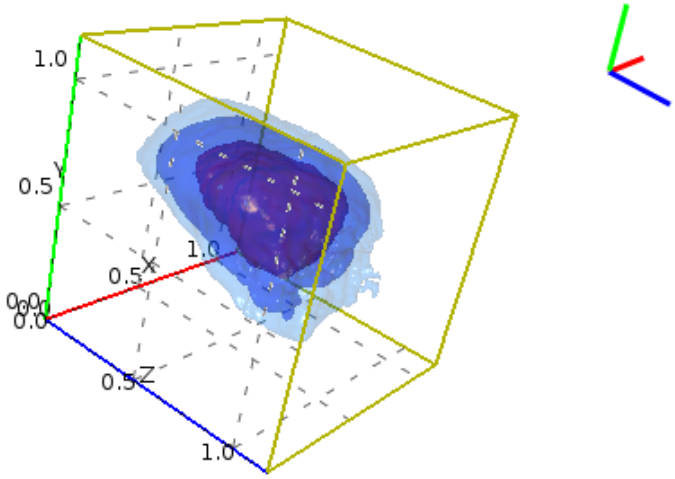

Fig. 2. 3D CDI reconstruction, with isosurface at 20%, 30% and 50% of the maximum amplitude. (Coordinates in um)

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In-situ compression of InSb micro-pillars under coherent X-ray diffraction

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During the last two decades, it has been shown that micro- and nano-objects exhibit different mechanical behavior compared to bulk counterparts, so-called "size effects" associated to increased elastic limit and/or different deformation mechanisms. For semiconductors, the size reduction is accompanied by a drastic change in mechanical response: while bulk semiconductors are brittle at room pressure and room temperature, they become ductile when in the form of micro-objects. The understanding of this transition is crucial for the micro-electronics industry, in continuous search for device miniaturization.

In this project (CharAdiff ANR-16-CE93-0006), we use coherent X-ray diffraction (CXRD) to detect and characterize the very first plastic events of a set of indium antimonide (InSb) micro-pillars, with height in the range 6-10.5 µm and aspect ratio of 3:1. The samples were characterized during in-situ micro-compression at two beamlines, Cristal (SOLEIL) and ID01, respectively with micro- and nano-focused beams. We will present the specific methodology needed for this study, due to the presence of a vastly diffracting pedestal, the size and weight of the compression machine, the artefacts potentially caused by the mechanical test. Then, we will show how this technique complete the already established ones (like electron microscopy and post-mortem CXRD) to characterize the mechanical properties of InSb micro-pillars.

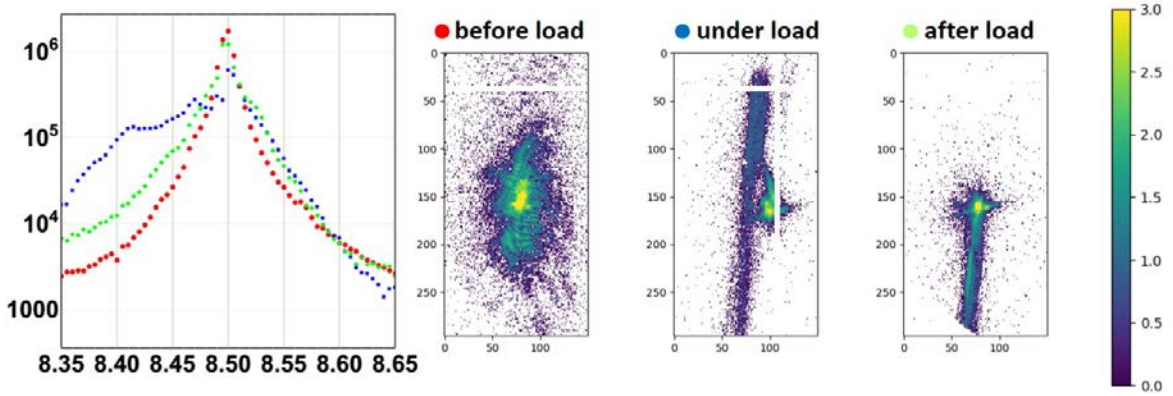

Figure 1: Left: Integrated intensity *vs* energy (in keV) of the InSb micro-pillar before (red), under (blue) and after (green) an applied stress of 550 MPa. Right: Two-dimensional diffraction patterns at 8.50 keV (log scale). Under load, the Bragg peak of the pillar dissociates from the one of the undeformed pedestal. A large elastic bending is monitored. After load, a streak evidences plastic defects in the micro-pillar.

Correlative 3D structural and chemical quantification of single human cells

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X-ray microscopy is increasingly used in biology, but in most cases only in a qualitative way. We present here a correlative X-ray microscopy approach suited for quantification of molar concentrations in cells at nanometre scale. By combining the elemental content provided by X-ray fluorescence microscopy and the morphology information extracted from X-ray phase nano-tomography, we determine the intracellular molarity distributions. This method was first demonstrated on a freeze-dried human macrophage cell to obtain the absolute elemental concentration maps of biologically relevant elements (Figure 1) [1]. The cell morphology results showed a very good agreement with atomic-force microscopy measurements. The correlative approach was extended to three dimensions and to the quantification in terms of local mass fractions on malaria-infected red blood cells [2]. While these proofs of principle were performed on freeze-dried cells, we pushed further the technique to cryo-preserved cells to better safeguard the cellular structure and elemental content. The quantification of major and minor elements, as well as the density, were extracted in the different organelles of frozen-hydrated cells. This work opens the way for in-situ single cell structural and chemical analysis down to sub-organelle level using exclusively synchrotron radiation techniques.

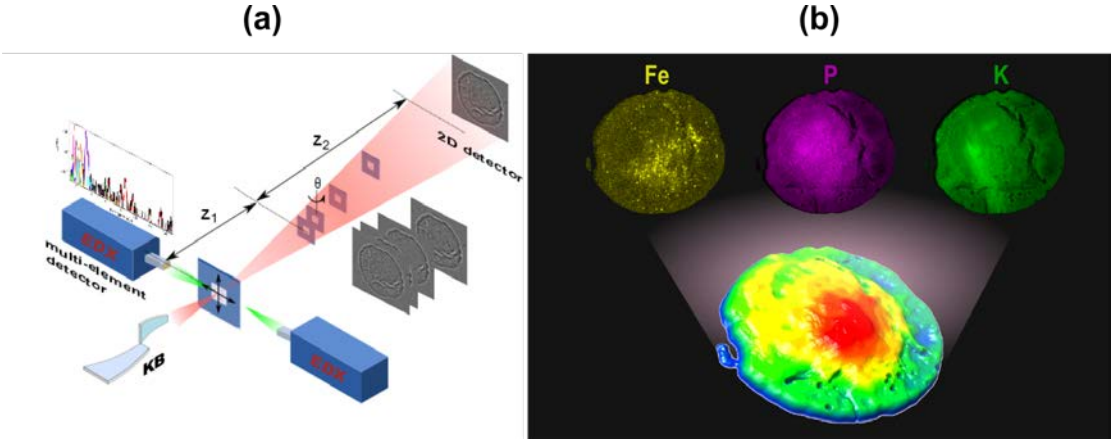

Figure 1: a) Acquisition scheme of correlative X-ray fluorescence and X-ray phase contrast imaging
b) Bottom: macrophage cell thickness derived from X-ray phase contrast nano-tomography. Top: elemental concentrations in the cell derived by the correlative method.

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Reconstruction of X-ray waveguide fluorescence holography for evolving nanostructures in thin films

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Fluorescence is the emission of light or radiation by certain substances as a result of absorbing incident radiation of a shorter wavelength. Applications of fluorescence such as in spectroscopy and microscopy do not utilize its steradian sensitivity because the directly emitted fluorescence is an isotropic outgoing spherical wave. However, an anisotropic power distribution of the fluorescence can be induced when the fluorescence is modulated by local environmental inhomogeneities near its emitting source. This concept has been explored as the X-ray fluorescence holography (XFH) on oriented crystalline samples, where local atomic structures can be reconstructed from fluorescence holograms with sub-atomic spatial resolution. Besides crystals, an electromagnetic waveguide can also modulate the internally emitted fluorescence, creating a concentric cone-shaped hologram when the fluorescence leaves the waveguide. In this work, we illustrate the principle of XFH for a waveguide and demonstrate that when applied to a thin film consisting of fluorescence substances, it becomes an *in-situ* and time-resolved imaging technique – X-ray waveguide fluorescence holography (XWFH) – for embedded nanostructures and their kinetics in the film.

Conventional XFH is done in either the normal or inverse mode. The holographic reconstruction in either mode is achieved via a back-propagation of the far field hologram in the framework of kinematic approximation. During the holographical reconstruction, these Fourier-transform based algorithms often find difficulties for actual XFH experimental conditions, causing ghost patterns or false atomic images due to the presence of non-kinematic effects such as mode mixing, self-interference, multiple scattering, extinction, and etc. In contrast, these effects are inherently significant in a waveguide for the XWFH, requiring the reconstruction to be performed in the framework of the dynamical theory. By applying an iterative holographical reconstruction algorithm to a mixed-mode XWFH carried out at both grazing-incidence and exit angles, we can take many advantages of the dynamical scattering effects and treat them as additional constraints for better convergence and reconstruction qualities.

Samples of gold nanoparticle monolayers embedded in supported polymer films were selected to illustrate the working principle of the mixed-mode XWFH. Buried nanostructures in thin films have often been measured with forward scattering-based techniques such as grazing-incidence small-angle X-ray scattering (GISAXS) and reflectivity. With XWFH, we were able to monitor the diffusion kinetics of the nanoparticles because the broadened nanoparticle distribution upon thermal annealing alters the waveguide conditions which were detected as the variation of the angular dependence of the fluorescence hologram. The advantages of performing XWFH on nanostructured thin films emerged when the dynamically reconstructed gold atomic number density distribution was compared to the result from the reflectivity and the structures from model fitting of the simultaneously collected GISAXS.

Pulsed magnetic field setup for temperature dependent dynamics studies

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Studies of externally excited ultrafast magnetic dynamics in condensed matter using coherent X-ray scattering techniques are subject of interest for decades. An important challenge in time-resolved magnetic studies concerns the ground-state symmetry breaking, and monitoring the recovery back through different transients. Combination of pump excitation and the possibility of probing both structural and electronic ordering in ultrafast XFEL experiments is a crucial aspect.

For these studies a pulsed magnetic field setup was designed to be employed at the Materials Imaging and Dynamics instrument (MID) at the European XFEL, based on previous version from ESRF [1]. The system has been successfully tested at beamline P09 at Petra III (figure 1). The liquid nitrogen cooled split-pair solenoid reaches a maximum field of 15 Tesla for total pulse duration of a few ms. The bunch clock of the FEL is used to synchronize a 4.5 MHz 2D detector (AGIPD), thus scattering is collected over the duration of the field pulse or offset with respect to the field. The synchronization of a single AGIPD module and B-field has also been successfully achieved at P09. The sample is cooled in an independent helium flow cryostat which is inserted into the bore of the magnet. The flow cryostat has a temperature range from ~5 to 250 K. Recently the first tests with this setup were carried out at MID with a Si(111) crystal.

Using this setup it is planned to study field-induced dynamics of the mesoscopic structure associated with the charge-density wave (CDW) that co-exists with the spin-density wave (SDW) in antiferromagnetic chromium (Cr). There has been a lot of debate about the connection between the SDW and the CDW that has a period of half the SDW and is either due to a magnetically induced lattice distortion or a purely electronic effect based on nesting of the Fermi surface [2,3].

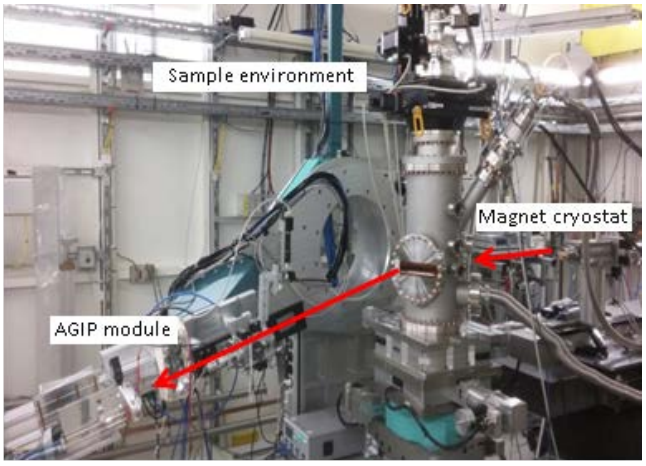

Figure 1: Experimental setup with the pulsed magnetic field apparatus mounted on a Huber diffractometer in EH2 at P09.

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ID01 in light of the ESRF-EBS

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The ID01 beamline was built to combine Bragg diffraction with imaging techniques to produce a strain and mosaicity microscope for materials in their native or *operando* state[1]. Conceived with the upgraded ESRF-EBS source in mind over the past four years it has been optimised to exploit the new source to the maximum, typically delivering coherent focused x-ray beams of 50nm – 1 micron in dimension [1,2].

The ESRF-EBS source will deliver increased coherent flux (x40), providing not only throughput and/or resolution gains but coherent diffractive imaging up to 35keV[3]. We will demonstrate the cutting edge available on ID01 today and our perspective on the opportunities made available by ESRF-EBS source.

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Dynamic critical scattering by XPCS in the ordering AuAgZn2 alloy

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The dynamics of the order fluctuations in the AuAgZn$_2$ close to the critical point (Tc~609 K) was observed by coherent x-ray scattering [1]. With the high beam intensity of the ID10 ESRF beamline and with the new pixel detector, the dynamics was measured with a few tens of millisecond resolution. The intensity connected to the diffuse scattering corresponding to fluctuations was unambiguously distinguished from the surface pretransitional ordering occurring in this system close to T$_c$. The variations of the fluctuation time with temperature and wave vectors were measured in this system belonging to the universality class of Ising second order transition with a non-conserved order parameter. The direct observation of the critical slowing down in the vicinity of the second-order transition led to an estimate of the dynamic exponent z=1.96(11), in rough agreement with theory (model ``A'' of [2]).

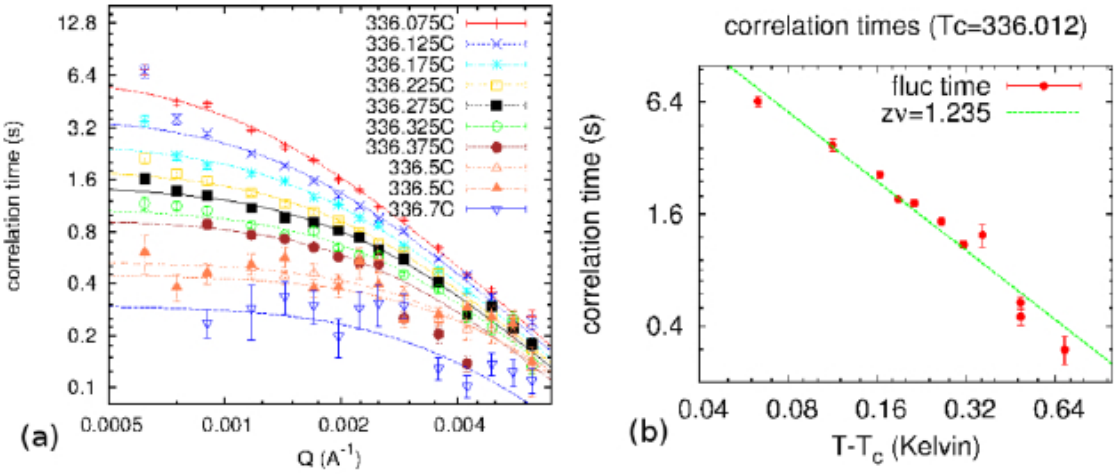

Figure 1: (a) Fluctuation times τ (q,T) for various temperatures and the Lorentzian fits. (b) Temperature variations of τ0(T). An estimate of the exponent yielded zν = 1.235(70).

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Cateretê, the coherent scattering beamline at Sirius, 4th generation Brazilian synchrotron facility

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The Cateretê beamline at Sirius, the new Brazilian synchrotron light source will be dedicated to coherent and time-resolved scattering experiments. It will provide unique capabilities, providing cutting edge research tools that are non-existent today in Brazil, like 3D imaging with nanometer resolution and X-ray photon correlation spectroscopy (XPCS) to study dynamics in hard and soft condensed matter and biological materials.

The SIRIUS storage ring will have a natural horizontal emittance of ϵ_x = 245 $pm\ rad$ and a vertical emittance (coupling 1%) of ϵ_y = 2.4 $pm\ rad$[1]. The Cateretê beamline will be equipped with a Delta undulator[2] allowing to tune the beam polarisation. The main three optical elements, vertical and horizontal focusing mirrors[3] and the four-crystal monochromator will be in a horizontal side-bounce configuration, optimising the stability, and delivering a fully coherent beam of 30 x 30 μm^2, with an energy ranging from 4 to 21 keV. The expected coherent flux is expected to reach 10^{12} ph/s at 4 keV. The experimental station will be located 88 meters from the source, followed by a 28 meters vacuum chamber hosting the Medipix (3k x 3k pixels[2]) in-vacuum detector. The sample environment is under commissioning and will enable to perform 3D-CDI, ptychography, BraggCDI and XPCS measurements. It will be equipped with an interferometry system, a cryogenic device for biological specimens and catalytic reactors for *in situ* studies[4,5].

In this poster presentation, I will present the main characteristics and new scientific potentialities of the Cateretê beamline which is expected to receive light by the end of 2019.

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The coherent applications instrument MID at European XFEL - commissioning and first user experiments

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The scope of the Materials Imaging and Dynamics (MID) instrument is material science using the unprecedented coherent properties of the X-ray beam of European XFEL [1]. Several X-ray scattering and imaging techniques are foreseen, for example Coherent Diffraction Imaging (CDI), X-ray Photon Correlation Spectroscopy (XPCS), and X-ray Cross Correlation Analysis (XCCA).

First lasing of the MID undulator was achieved in May 2018, followed by the first beam in the MID optics and experimental hutch in October and December 2018, respectively. From March to June 2019, the first early user experiments were conducted, using techniques such as X-ray holography, XPCS or XCCA.

We will present the concept and setup of the MID instrument, which was designed to offer many different experimental configurations and enable high quality coherent X-ray experiments. We outline the commissioning of the instrument during the first half of 2019 and show results of the commissioning and early user experiments conducted at MID.

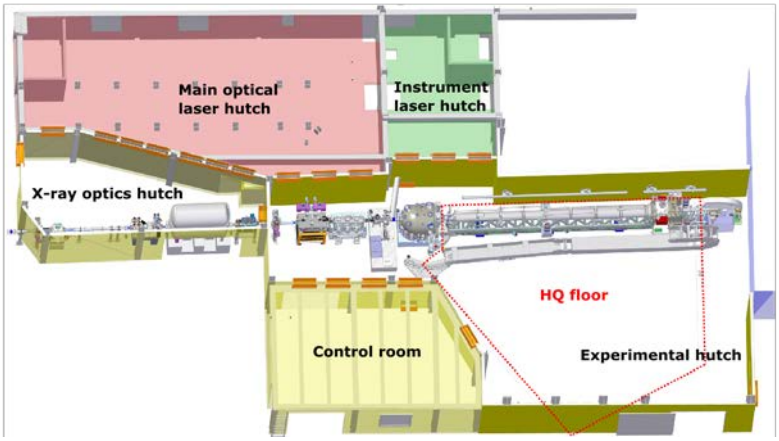

Figure 1: Top view of the MID instrument and its enclosure.

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Multi-order imaging with multilayer zone plates

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Diffractive optics such as Multilayer Laue Lenses and Multilayer Zone Plates (MZPs) have emerged as nano-focusing optics in the hard x-ray regime around 8 keV and higher. In “traditional” set-ups, the negative and higher diffractive orders are blocked out by an Order Sorting Aperture (OSA); here we show that multi-order holographic datasets in a scanning set-up can be disentangled using a ``software-OSA". This allows for more flexibility in the experiment, a zoom-in capability at different field-of-views, and the correlation of different contrast mechanisms.

We show how to extract high-resolution differential phase contrast information on a nanometre scale combined with a micro-metre sized phase-reconstruction; see the figure for an example on nanowires.

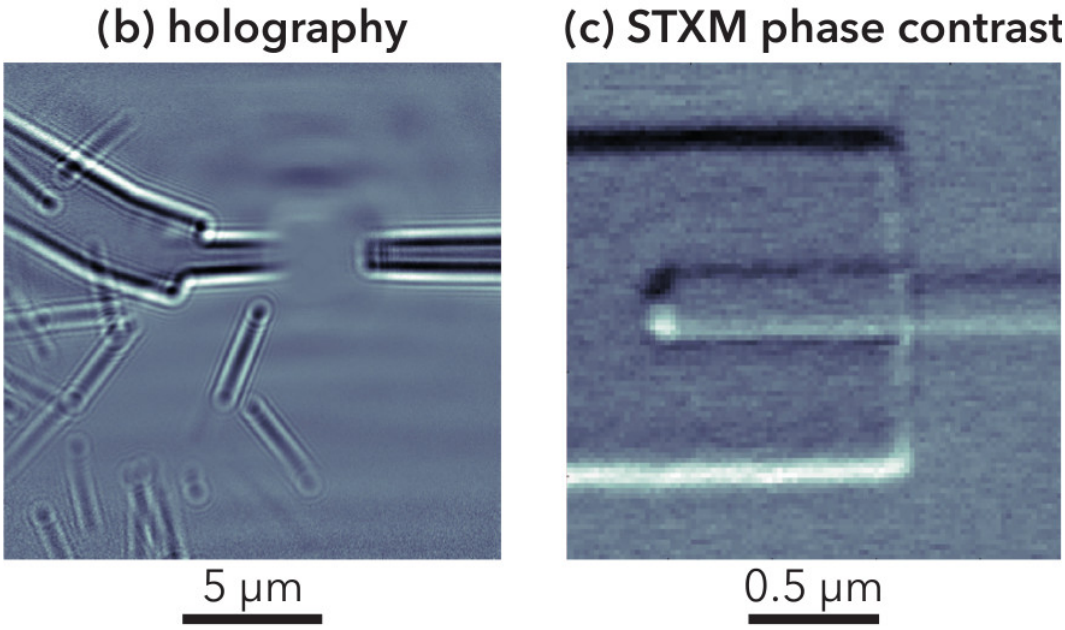

Figure 1: Holographic and differential phase information extracted from a single dataset using an MZP optic at 13.8 keV X-ray energy.

Focus characterization of the NanoMAX Kirkpatrick–Baez mirror system

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The focusing and coherence properties of the NanoMAX Kirkpatrick-Baez mirror system at the fourth-generation MAX IV synchrotron have been characterized. The direct measurement of nano-focused X-ray beams is possible by scanning of an X-ray waveguide, serving basically as an ultra-thin slit. In quasi-coherent operation, beam sizes of down to 56 nm (FWHM, horizontal direction) can be achieved. Comparing measured Airy-like fringe patterns with simulations, the degree of coherence $|\gamma|$ has been quantified as a function of the secondary source aperture (SSA); the coherence is larger than 50% for SSA sizes below 11 µm at hard X-ray energies of 14 keV. For an SSA size of 5 µm, the degree of coherence has been determined to be 87%.

The thick green line in the left figure shows the simulated intensity profile for a finite SSA size of 15 µm convolved with the WG channel. The right figure shows the experimental contrast values as orange circles at different SSA sizes between 5 µm (quasicoherent) and 50 µm (full flux). The thick red curve shows the corresponding WG-convolved simulated data. Experimental and simulated values agree within 2σ, and the experimental values reflect the oscillating behaviour of the contrast with minima at 18 µm and 36 µm.

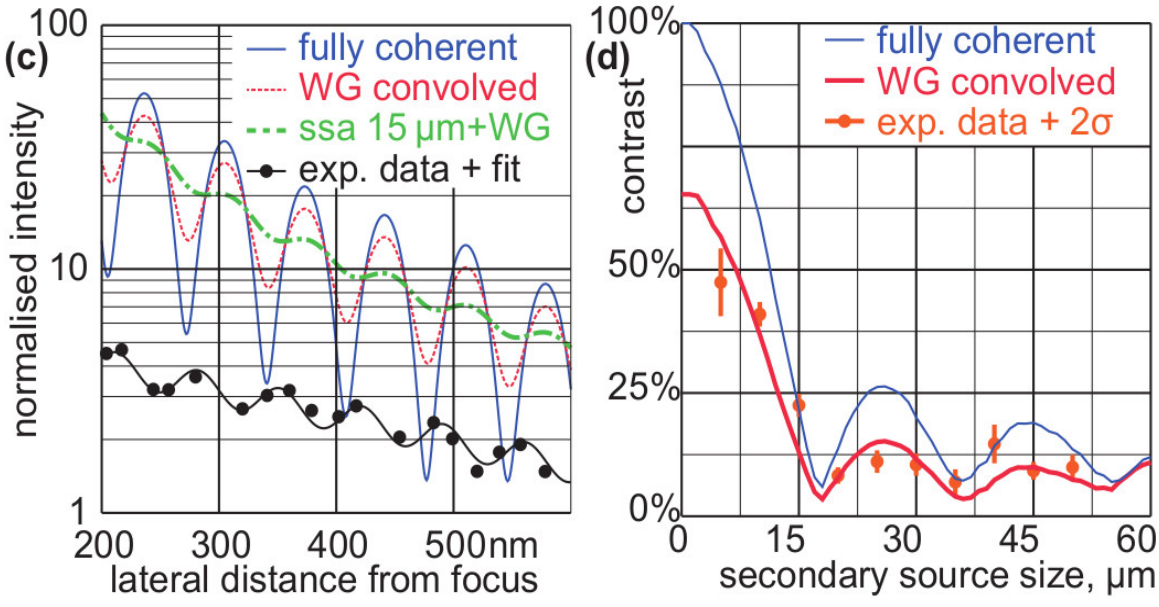

Figure 1: Simulated and measured fringe contrast to determine the coherence properties of the NanoMAX focussing system. Left: Fringe visibility at a fixed secondary source aperture; right: degree of coherence as a functino of secondary source aperture.

Investigation of osteons properties in human cortical bone using X-ray phase micro-CT

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Cortical bone, which plays a major role in the biomechanical properties of bone, has a complex multi-scale structure. Due to the bone remodeling process, cortical bone is made of osteons and interstitial tissue having different material properties. There are however, still few 3D quantitative data regarding bone osteonal systems. This lack of data can be explained by the difficulty to distinguish osteons from interstitial tissue using standard 3D imaging techniques [1]. Since phase contrast techniques permit to increase image sensitivity, they are good candidates to analyze osteons [2].

In this work, we used phase micro-CT at the sub-micrometer scale for the quantitative analysis of osteons in human cortical bone and their relationships to the distribution of osteocyte lacunae.

Rectangular cortical bone samples prepared from eight female donors radii (50 to 91 y.o.). were imaged on beamline ID19 at the European Synchrotron Radiation Facility (ESRF, France). Phase contrast was obtained by propagation with a sample-to-detector distance set to 40 mm and an effective pixel size on the detector of 0.7 µm. We used a "pink beam" with an energy of 31 keV. Phase retrieval was performed using Paganin's method. The Haversian canals, osteocyte lacunae and micro-cracks were segmented automatically using a home-made method [3] and the osteons were segmented manually using Avizo software.

Figure 1 illustrates one phase micro-CT slice in cortical bone and a 3D display of the segmented image. Our quantitative results showed differences of lacunar density between osteonal and interstitial tissue and a higher volume fraction of micro-cracks in the interstitial tissue.

X-ray phase micro-CT can be used to obtain new information about the properties of osteons and their role in the biomechanical resistance of bone tissue.

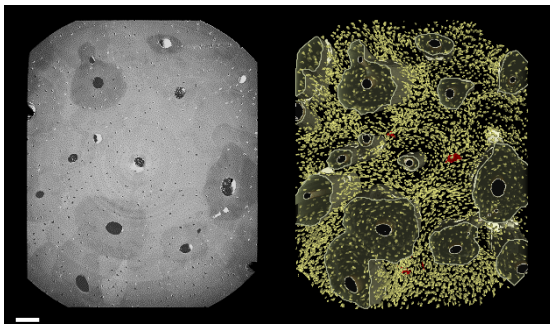

Figure 1: Phase micro-CT image of radius cortical bone. left: original slice (osteons appear darker) ; right: 3D display of the segmented volume (osteon border: green, lacunae: yellow, micro-crack: red). Scale bar 100 µm.

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Glassy and liquid metals, from microscopic to macroscopic dynamics

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The relaxation dynamics of glass-forming metals control some of their key mechanical properties. The dynamics of the glassy state control the change from homogeneous to heterogeneous plastic deformation when applying deformation at different velocities, as well as it determines the physical aging behaviour that can produce a ductile to brittle transition. At higher temperatures, in the glass transition region and the supercooled liquid state, the dynamics control the rheological behaviour and the ability of thermoplastic forming. X-ray Photon Correlation Spectroscopy (XPCS) gives access to the microscopic dynamics of glasses form the atomic length scale up to the nanometer scale. This is the range of scales in which it is expected that the cooperative dynamics of supercooled liquids and glasses can be observed. On the other hand, mechanical spectroscopy, creep experiments and stress relaxation give access to the macroscopic dynamics [1]. An important remaining task still to do is to theoretically connect the microscopic and macroscopic dynamics, thus unveiling the atomic and nanometer-scale mechanisms of the macroscopic behaviour.

This poster presents mechanical relaxation and XPCS data obtained for various glass-forming alloys, namely $Pd_{40}Ni_{40}P_{20}$, $Pd_{40}Ni_{10}Cu_{30}P_{20}$ and $Zr_{46.75}Ti_{8.25}Cu_{7.5}Ni_{10}Be_{27.5}$, comparing the times and shapes of the macroscopic and microscopic relaxation functions in the glassy, glass transition and supercooled liquid regions, thus covering relaxation times from the order of thousands of seconds to the order of few seconds. At temperatures above the glass transition, the macroscopic dynamics are coincident in shape and time with the microscopic dynamics measured at the maximum of the structure factor. The distribution of times at this length scale is reproduced in the stress relaxation function, with similar values of stretched exponents. On the other hand, in the glassy state, the macroscopic relaxation is dependent of the level of external force applied in the experiments, changing from partial to complete relaxation at the same temperature but different levels of stress. In this case the microscopic relaxation time seems to control only the partial relaxation observed when applying low stresses. Furthermore, the broad spectrum of relaxation times observed macroscopically in this region is not reproduced in the microscopic dynamics where compressed relaxation functions are always observed.

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Bagasse nano-structure modifications induced by hydrothermal pretreatment revealed by CXDI

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In recent decades, the interest in the search for alternative energy sources has stimulated the use of plant bagasse generated as a product from ethanol production, such as the sugarcane waste in Brazil[1]. This material can be used as source of different sugars, for example, the cellulose, which can also be employed as a raw material in energy, paper and food industrial sectors. To extract cellulose, the bagasse must be submitted to chemical or physical processing such as the hydrothermal pretreatment[2]. The exploitation efficiency of the sugar extraction might depend on nanometric structural changes caused by the pretreatment[3]. Thus, we performed three-dimensional coherent X-ray diffractive imaging (3D CXDI) measurements of dry raw sugarcane (*in natura*) and hydrothermally pretreated bagasse samples, at ID10 beamline at ESRF (France), to verify changes in the porous network. In this presentation, we will propose a new methodology to prepare ~5 µm fragments of sugarcane bagasse and how the final ~30 nm spatial resolution three-dimensional allowed to perform accurate image analysis (segmentation). The CXDI experiments were a direct method to depict the morphological disruption of the fibrillar character from cellulose and the modifications in bagasse porosity. The understanding of porous network modifications is crucial to verify the material access to hydrolytic enzymes used in the further steps and is compulsory to determine and evaluate the pretreatment efficiency.

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Soft*i*MAX - A new soft X-ray microscopy and coherent imaging beamline at the MAX IV facility

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Soft*i*MAX is a new beamline for soft X-ray spectro-microscopy currently under construction at the new MAX IV synchrotron facility in Lund, Sweden [1]. It is situated at the 3 GeV ring at MAX IV, and will provide a very high average coherent flux owing to the low emittance properties of the ring [2]. The Soft*i*MAX project consists of two branch lines, utilizing different focusing solutions and catering to a variety of imaging methods. As the construction of the beamline is nearing completion, we present the design and an outlook on the capabilities of the experimental stations.

In overview, the available photon energy range of Soft*i*MAX is 275 to 2500 eV, with full polarization control up to approx. 1650 eV. The energy resolution will lie between 1000 and 15000 E/dE, depending on the grating and energy used, and the flux on sample in the main branch end-station is ca. 2×10^{10} ph/sec at 275 eV and ca. 2×10^{9} ph/sec at 2500 eV with a 1200 l/mm grating and a 20 nm-FZP.

The main branch of Soft*i*MAX will host an end-station optimized for scanning techniques such as Scanning Transmission X-ray Microscopy (STXM) and diffraction imaging (ptychography [3]). Accordingly, the x-ray beam will be focused using Fresnel Zone Plates (FZP) to a beam size on the sample below 100 nm to allow for small illumination footprint (STXM) or a larger beam size to ease methods that require overlapping illumination (ptychography). STXM will be the first method offered at Soft*i*MAX in early 2019, and the STXM branch is envisioned to support a wide range of sample types, including material science, environmental science, life science and biology.

The second branch of Soft*i*MAX will provide a larger spot size than the main branch, and higher flux, by using Kirkpatrick-Baez focusing optics. The envisioned spot size on the sample is here 20 μm, to support methods that require a fully coherent extended beam, such as Fourier Transform Holography, which is the principal mode of operation for this branch. The corresponding end-station will be optimized for the studies of magnetic thin films using magnetic dichroism, but is still in the planning stage.

Soft*i*MAX is part of the Imaging group at MAX IV, and embedded in a wider infrastructure for sample preparation and data analysis, including a dedicated com-puter cluster and in-house support for spectral analysis and phase retrieval of diffraction data.

First user operation on the main branch is expected to start in early 2020.

The Soft*i*MAX project is funded by the Swedish Reaserch Council.

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A new X-ray reflecto-interferometer based on refractive optics

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Compound refractive lenses (CRLs) have been widely used for more than 20 years on leading synchrotrons for X-ray beam transport, collimation and focusing [1-2]. The area of CRLs applications was extended to the field of coherent diffraction and imaging techniques, Fourier optics and interferometry.

A new amplitude division X-ray interferometer operating in reflection mode was proposed and realized for the study of thin-film structures [3]. The reflecto-interferometer employs a CRL to produce a converging fan of radiation, incident onto a sample surface, and a high resolution CCD detector to simultaneously collect the reflecto-interferogram over an angular range matching that of the incident fan. The interference pattern, which is in fact specular X-ray reflectivity, is recorded in a single shot using very simplified experimental setup without the need to rotate the specimen or detector. Another feature of the proposed reflecto-interferometer is the spatial resolution, which achieved due to focusing by CRLs, allowing to probe different regions on the film.

The capabilities of the new reflecto-interferometer were demonstrated at the ESRF ID06 beamline using X-rays from 10 to 20 keV. The Si_3N_4 membranes of thicknesses in the range from 100 to 1000 nm were studded. Experimentally obtained reflecto-interferograms are in good agreement with calculated ones, and the distance between fringes correspond to the thickness of the tested membranes. While the interference pattern in rather wide angular range is recorded in one shot, the proposed approach has a very good temporal resolution, which is limited to a few milliseconds, depending on the X-ray flux and the sensitivity of the detector. The temporal characteristics of the interferometer were demonstrated by studying the radiation damage of the resist layer. The high spatial resolution of the interferometer was revealed on interferograms recorded from the gold strip deposited on the Si_3N_4 membrane. It was shown that the interference pattern is very sensitive to deviations in the thickness of the layer, resulting in ability to determine very accurately not only the average thickness of the gold layer, but also to reconstruct the cross-section profile of the gold layer.

The recording of the interference pattern in a single shot allows an express analysis of films, which is especially important for radiation sensitive materials, including organic and biological films. The spatial resolution property is very significant possibility to study samples with small lateral size, and is also necessary for the local analysis of the film. It should be emphasized that the put forward reflecto-interferometer can be employed for characterization of films with thickness from 20 nm up to few microns. In addition, this reflecto-interferometer can be easily adapted for use with laboratory X-ray sources.

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Measuring speckle Q-Q correlations

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A sequence of papers has previously been presented on the in-situ combined stress-strain and Xray Photon Correlation Spectroscopy (XPCS) measurements using filled elastomers [1-3]. Here, a new analysis of some of this data is presented in which the systematic shifts of the speckles are analyzed allowing for flow patterns of the particle relaxation in the elastomer can be inferred. This information is obtained from cross-correlations of speckle patterns $\langle I(Q,t)I(Q+\delta Q,t+\delta t)\rangle$.

The samples consisted of an elastomer (Ethylene Propylene Diene Monomer, EPDM, rubber) filled with carbon black (N330) or hydoxylated pyrogenic silica (Aerosil 200, DEGUSSA) [4-6]. In our measurements, the one millimeter thick plates are punched out to the classical dumb-bell shape (width = 4 mm). An example flow pattern is shown in Figure 1.

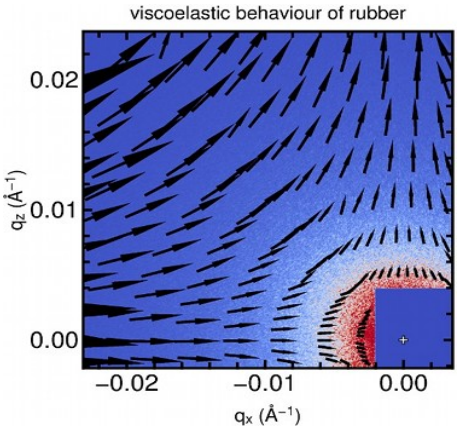

Figure 1: The velocity field of an ethylene-propylene polymer with 16% silica filler, at 60% elongation, 3000s into the stretch. Using coherent xrays, the small angle xray scattering (SAXS) pattern is modulated by a speckle pattern. The deformation of the particle positions under flow leads to a shift in the speckles. The velocity gradient shown is extracted from the change in speckle positions for patterns 10.5s apart. The arrows show that shift scaled by a factor of 200 superimposed on the coherent SAXS pattern. The xray beam was 20 microns by 20 microns in cross section.

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X-ray thin-film interferometry technique using an X-ray microfocus laboratory source

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X-ray reflectometry (XRR) is actively used for thin-film and multilayer systems internal structure study. Nevertheless, this method has a number of limitations associated with high requirements to the sample surface quality and geometric parameters. The advancement of new optics for X-ray laboratory and synchrotron sources increased opportunities for the development of X-ray diagnostics techniques, including reflectometry. In this work, we demonstrate a new X-ray interferometry technique based on compound refractive optics (CRL) for thin-film structures study.

Nowadays, X-ray refractive optics is the most dynamically developing optical field [1-2]. Compound refractive lenses quickly gained popularity among users of synchrotron sources due to their operation simplicity and the applicability in a wide range of energies [3-4]. The combination of modern optical elements with X-ray radiation sources opens up new possibilities for thin-film structures diagnostics, allowing to overcome the limitations of classical laboratory techniques.

The idea of the recently proposed X-ray interferometry technique is to use a simplified experimental setup in which a focused X-ray beam is reflected from the flat plate surfaces creating an interference pattern in a wide angular range without the need to rotate the specimen or detector [5]. The applicability of this technique has been demonstrated using the MetalJet Excillium micro-focus laboratory source, which is a part of Synchrotron-Like facility (IKBFU, Kaliningrad, Russia). A series of interference patterns for thin-film membrane thick of 500 um were observed.

The new X-ray reflecto-interferometry technique opens a wide range of opportunities for the analysis of thin-film and multilayer systems. This technique can be realized using both laboratory and synchrotron radiation x-ray sources. Also, it provides advantages over the conventional X-ray reflectometry because it allows for research with a fundamentally new temporal and spatial resolution.

This research was supported by Russian Science Foundation (Project No. 19-72-30009)

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Placing quantum dots in 3D photonic crystals and finding them back

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It is a major outstanding goal in Nanophotonics to precisely place quantum emitters inside a three-dimensional (3D) metamaterial. This offers control over cavity QED, spontaneous and stimulated emission, and non-linear optics [1]. Theory says that emission (e.g. from quantum dot) varies spatially on 100s nm scale [2]. Thus, we want to place emitters with a precision $\Delta x < 100$ nm. We developed a chemical toolbox to position quantum dots on 10 nm thick polymer brush layers inside 3D Si nanostructures [3]. Our photonic crystals have the 3D inverse woodpile structure (Fig. 1(A)) that exhibits a broad 3D band gap. The crystal are made by CMOS-compatible methods using deep reactive ion-etching through tailored masks. Here we want to learn if the quantum dots are at the right place?

Since nanophotonic materials are necessarily opaque, optical microscopy has insufficient penetration depth and limited resolution. SEM has plenty spatial resolution, but only the surface is viewed (Fig. 1(A)). Thus X-ray tools are ideal, especially X-ray fluorescence tomography, done at the ESRF beamline ID-16NI. We collect data while rotating the crystals from 0 to 180°. Projection maps are obtained at every angle followed by standard tomographic reconstruction to obtain the 3D atom density distribution with 50 nm spatial 3D resolution for each chemical element.

Fig. 1(B) shows a projection map of the lead atoms (PbS quantum dots) in one crystal. The 3D volume after reconstruction reveals two sets of pores running in Z and X-directions, matching the design (Fig. 1(A). The structure is periodic with lattice parameters that match the design. Quantum dots are located throughout the whole crystal volume. Their position correlates well with elements characteristic of the polymer brush layer. We conclude that X-ray fluorescence tomography is superb to solve many questions on 3D optical metamaterials, including cavity superlattices, physically unclonable keys, and precise positioning of emitters as qubits and for enhanced lighting efficiency. We look forward to the novel opportunities presented by speckle and coherent X-rays in the new ESRF!

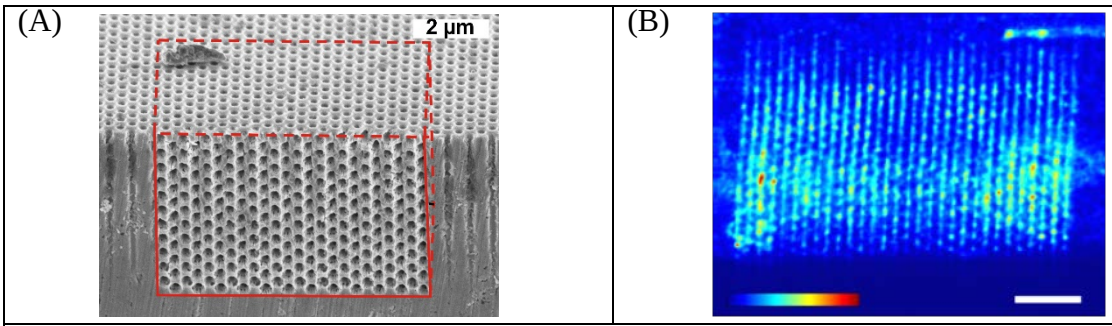

Fig. 1. (A) SEM image of the surface of a 3D Si inverse woodpile photonic crystal. The estimated extent is shown by dashed lines. The 3D crystal is surrounded by a large 2D array of pores that is first etched in the Z-direction. **(B)** Projection map of the number of Pb atoms per pixel (50 x 50 nm^2) integrated along the propagation direction of the beam, for an orientation as in (A). The color scale is the areal atomic density ranging from 0 to 420000 Pb atoms per pixel. The scale bar is 2 µm.

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X-ray imaging of functional three-dimensional nanostructures

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Three-dimensional (3D) nanostructures are drawing major attention for advanced functionality in nanophotonics [1], photovoltaics, novel 3D integrated circuits and flash memories. The functionality of such nanostructures are set by their complex internal structure. Since real nanostructures inevitably differ from the design, the observed functionality differs from the expected one. It is thus critical to know the structure of a 3D nanomaterial and verify how well it matches the design. Ideally such a technique leaves no traces of the inspection to leave the nanostructure fully functional and ready for integration. We thus introduce traceless X-ray tomography (TXT) as a new nanotechnological methodology to study functional nanostructures on thick substrates "as is".

Usually, a new nanostructures is inspected by SEM. But only the external surface is then viewed while the 3D structure remains hidden (Fig. 1A). SEM may be supplemented with ion beam milling to cut part of the structure away. This approach is undesirably destructive and irreversible. To achieve nanometer spatial resolution, we employ X-ray holographic imaging [2] that was done at the ESRF beamline ID16A-NI. We study Si 3D photonic band gap crystals made by CMOS-compatible means (Fig. 1A) [4]. These nanostructures are powerful tools to control the propagation and the emission of light [3].

Fig. 1A shows a bird's-eye view of the reconstructed sample volume of the 3D photonic crystal shown in Fig. 1. The Y Z top face shows the surface of the X-directed pores, similar to the SEM surface in Fig. 2A. The pore alignment determines the 3D structure and is a crucial step in the nanofabrication. In practice, the alignment is controlled by the etch mask for each pore array and by the directionality of the etching. We find that pores are running in the Z direction, whereas in the XY front face, pores are running in the X direction, matching the 3D design of the inverse woodpile structure. Notably the fabricated structure stays fully functional after X-ray imaging as it does not undergo any preparation step. We look forward to new opportunities using speckle imaging with coherent X-rays.

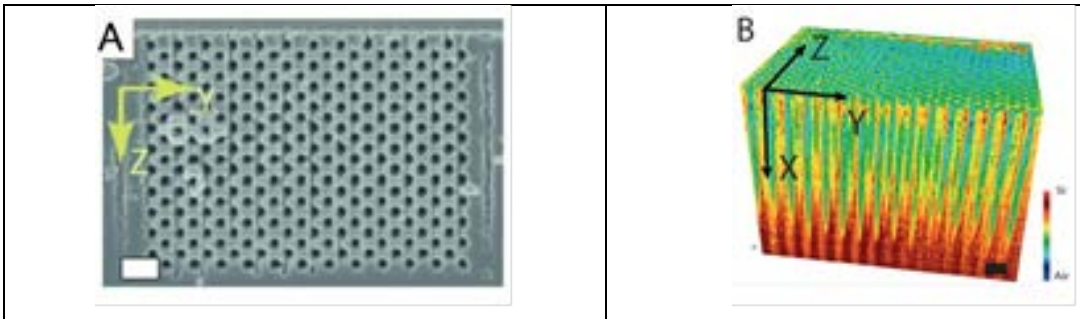

Fig. 1. **(A)** SEM image of the ZY-surface of a 3D Si inverse woodpile photonic crystal from Ref. [3]. **(B)** Bird's-eye view of an inverse woodpile crystal reconstructed from X-ray tomography with 55 nm resolution. The colour scale is the 3D material density $\rho(X,Y,Z)$ interpolated between air and Si.

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Large beam X-ray photon correlation spectroscopy

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The Coherence Beamline P10 at PETRA III is dedicated to coherent X-ray scattering experiments using X-ray Photon Correlation Spectroscopy (XPCS) and Coherent Diffraction Imaging (CDI) techniques.
Mostly, the beamline operates in the energy range of 5-17 keV. It consists of two 12 m long experimental hutches (EH1 & EH2) which house various experimental setups. Here, we present the large beam XPCS setup at P10. In this configuration, the sample position is situated in the first experimental hutch (EH1), while the detector is positioned at the end of the second hutch (EH2), which results in a sample to detector distance of around 21.3 m. This long pathway allows it to use a large fraction of the horizontal coherent flux in an unfocused X-ray beam, while providing a fairly strong speckle visibility at 8 keV using a pixel size in the range of $(75\ \mu m)^2$. The setup is therefore ideally suited for radiation sensitive samples such as most complex liquids, as the flux per sample area can be considerably reduced using a comparatively large X-ray beam.
At a photon energy of 8 keV, the minimum accessible q is around $2x10^{-4}$ $Å^{-1}$. To illustrate the possibilities for XPCS in this configuration, recent experimental results will be displayed.

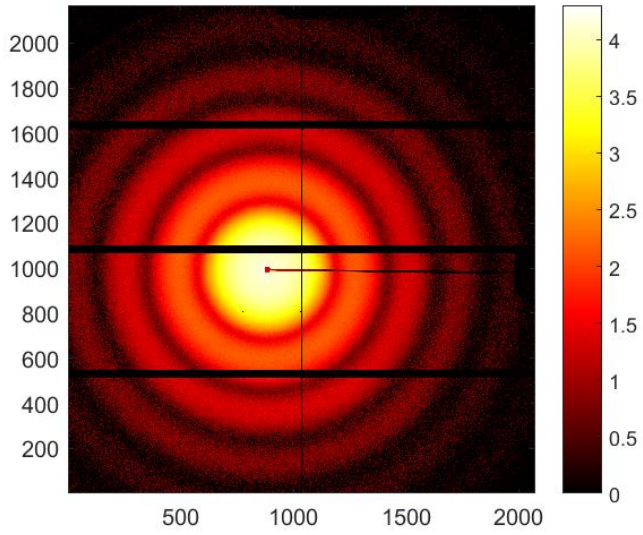

Figure 1: Scattering pattern of a nanoparticle suspension with a mean particle radius of 100 nm as detected by an Eiger X4M detector with a 155x162 mm^2 sensitive area.

Mapping data between sample and detector conjugated spaces in Bragg coherent diffraction imaging

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Bragg Coherent X-ray Diffraction Imaging (BCDI) is a non-destructive, lensless method for 3D-resolved, nano-scale strain imaging in micro-crystals. A challenge, particularly for new users of the technique, is accurate mapping of experimental data, collected in the detector reciprocal space coordinate frame, to more convenient orthogonal coordinates, e.g. attached to the sample. This is particularly the case since different coordinate conventions are used at every BCDI beamline, thus reconstruction algorithms and mapping scripts composed for individual beamlines are not readily interchangeable. We introduce a MATLAB BCDI experiment simulation with a plugin script that converts all beamline angles to a universal, right-handed coordinate frame, making it possible to condense any beamline geometry into three rotation matrices [1]. These matrices are implemented in MATLAB scripts that allow the user to map between various BCDI-related spaces, shown in Fig. 1, across different beamlines.

The ability to easily transform data between different spaces will be key for popularising BCDI, especially following the Extremely Brilliant Source (EBS) upgrade at the ESRF. The completion of the EBS upgrade at ESRF will see increased coherent flux, placing BCDI in a position to become a widespread imaging technique and microscopy tool for a general user community. In addition to simplifying space mapping, the scripts are also important for developing more robust phasing strategies, e.g. using projections from electron microscopy for phasing, and provides sample analysis flexibility, e.g. viewing the object in sample space or lab space. These tools are intended to serve as an initial toolkit for users to apply to various frameworks in their chosen disciplines. They can be downloaded from https://github.com/Hofmann-Group.

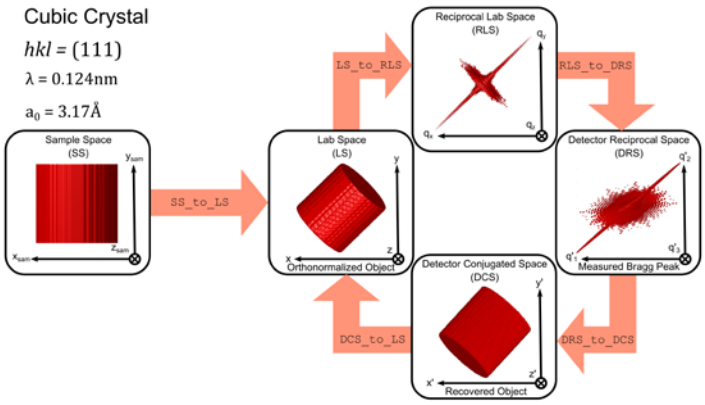

Figure 1: BCDI-related spaces in the MATLAB simulation [1] for a simulated cubic crystal cylinder at *hkl* = (111), a_0 = 3.17 ˚A and λ = 0.124 nm. MATLAB script filenames that convert from one space to another are shown inside the arrows.

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3D Bragg CDI of five-fold twinned Au nanoparticles

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Pentagonal bipyramid gold crystals have attracted the interest of scientists for many decades due to their apparent five-fold symmetry [1]. Closer inspection reveals that the nanocrystals must be composed of five distinct tetrahedral units separated by twin planes in order to exist [2]. Joining such five tetrahedral domains along their [110] axis leaves however a gap of 7.35° so that a solid could in principle not be formed. The mechanism of the stabilisation of these nanocrystals has been subject of debate and is expected to involve a complex stress state [3].

Here we use a combination of Laue micro-diffraction and 3D Bragg Coherent Diffraction Imaging (CDI) to shed light on the crystallography of pentagonal bipyramid Au crystals of 600nm in size (Fig. 1a). We show how the predicted five tetrahedral domains can be individually indexed in the Laue data and display features characteristic of a Σ3 twinned structure. We further discuss the complex peak shape visible in both the Laue and CDI data (Fig. 1b) and how this relates to the strain stabilising the nanoscrystal shape. Finally, we discuss the ongoing efforts to phase the CDI data, which will eventually allow us to retrieve the full 3D strain tensor of the nanoscrystal revealing the unknowns of its complex nature.

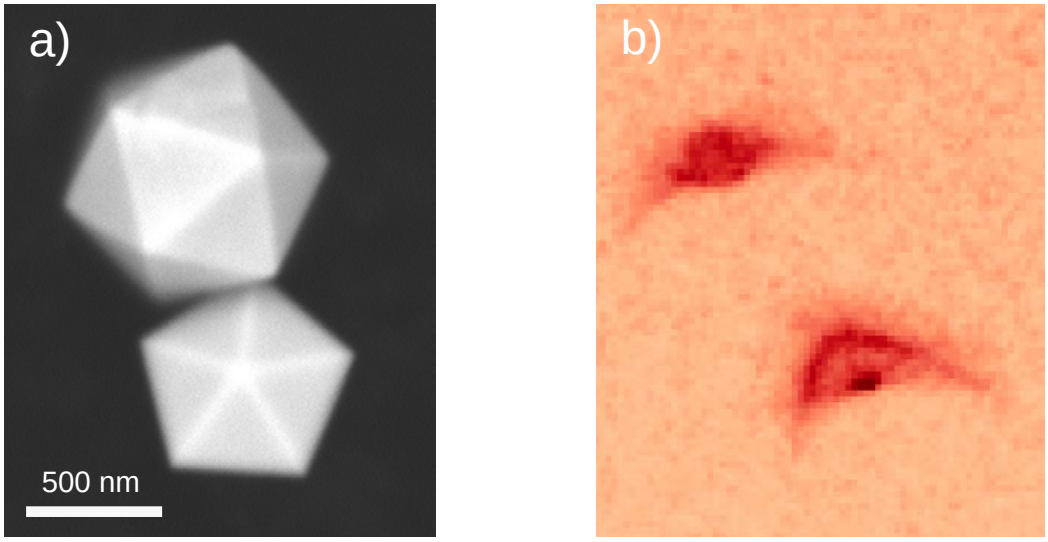

Figure 1: in a) a pentagonal bipyramid crystal is visible on the bottom left corner of the SEM image. The crystals are deposited on an Si substrate. In b), the complex shape of the Laue microdiffraction peaks is visible.

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