The annual event dedicated to the ESRF user community and to user science

ESRF
USER MEETING

6-8 February 2023

6 FEBRUARY
10 Tutorials for users

7 FEBRUARY
Plenary Session
Commercial Exhibition
Poster Session

8 FEBRUARY
3 User-Dedicated Microsymposia

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ESRF USER ORGANISATION

ESRF The European Synchrotron

EPN Science Campus

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38000 Grenoble

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User Meeting 2023

Monday 6 February – Tutorials

T1. BAG Meeting
T2. Getting the most from new in plate and plate to beam modalities on MASSIF-1
T3. Volume image analysis of tomographic data
T4. Science, communication and social media
T5. Data reduction for scattering experiments
T6. Introduction to XAS
T7. EXAFS data analysis
T8. Ab initio simulation of X-ray absorption spectroscopies using FDMNES
T9. XPCS: X-ray Photon Correlation Spectroscopy
T10. Coherent imaging analysis with PyNX: CDI (small angle and Bragg), ptychography, holotomography

Tuesday 7 February – Plenary Session

Venue: ESRF Auditorium

Administrative Assistants: Catherine Blanc – Agathe Chebre – Sonya Girodon

Tel: +33 (0)4 76 88 23 58 / 29 80 / 28 80 – usermeet@esrf.fr

Wednesday 8 February – Microsymposia

UDM1 Environmental sciences: challenges and opportunities under a new era of synchrotron light
Venue: **IBS Seminar Room**
contact: udm1-um23@esrf.fr
Administrative Assistant: Eleanor Ryan
Tel: +33 (0)4 76 88 19 92

UDM2 Tomography at BM18
Venue: **ESRF MD-1-21**
contact: udm2-um23@esrf.fr
Administrative Assistant: Eva Jahn-Feppon
Tel: +33 (0)4 76 88 26 19

UDM3 Operando science of functional energy conversion, storage materials and devices
Venue: **ESRF Auditorium**
contact: udm3-um23@esrf.fr
Administrative Assistant: Claudine Romero
Tel: +33 (0)4 76 88 20 27

ESRF USER MEETING 2023

OVERALL PROGRAMME

MONDAY 6

10 TUTORIALS

8:15 – 9:00 Registration
Central Building
WELCOME COFFEE

9:00 – 12:00 TUTORIALS

LUNCH BREAK

13:00 – 14:00 Registration
Central Building

14:00 – 18:00 TUTORIALS

18:30 – 20:30 *BUFFET DINNER*
In Marquee

TUESDAY 7

PLENARY SESSION

8:15 – 9:00 Registration
Central Building
WELCOME COFFEE

9:00 Opening & Welcome
9:05 Kristina Djinović-Carugo
9:50 Kirsi Lorentz

COFFEE BREAK

11:00 – 12:30 ESRF-EBS Facility & Director's Report

LUNCH BREAK

14:00 Paul Loubeyre
14:45 Sandrine Lyonnard

COFFEE BREAK

16:00 Poster Clips
17:00 Young Scientist Award
17:50 Conclusion

18:00 – 22:00 *POSTER SESSION & COCKTAIL DINNER*
In Marquee

WEDNESDAY 8

3 USER-DEDICATED MICROSYMPOSIA

8:15 – 9:00 Registration
At Event Venue

UDM1
IBS Seminar Room

8:40 – 17:00 Environmental sciences: challenges and opportunities under a new era of synchrotron light

UDM2
ESRF MD-1-21

9:00 – 16:00 Tomography at BM18

UDM3
ESRF Auditorium

9:00 – 16:00 Operando science of functional energy conversion, storage materials and devices

END OF MEETING

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Practical information

Badge information

You have been given a **BADGE** at the site entrance: **this badge is strictly personal** and due to the strengthened security measures, **you MUST wear your badge at all times on site over the whole period of the event**. It is your pass for the site entrance and, on site, for the Guesthouse and the canteen. **Please note that access to the Experimental Hall is strictly forbidden**.

Lunches

Lunches **are served** from

11:30 - 13:30

ground floor - restaurant

11:30 - 13:00

1st floor - restaurant

Please present your **BADGE** to the cashier.

Monday 6 February

Buffet Dinner

18:30 – 20:30

under the marquee

Tuesday 7 February

Poster Session & Cocktail Dinner

18:00 – 22:00

under the marquee

Hotels in the center of Grenoble

HOTELS

1 Ibis Grenoble Gare
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5 Maison Barbillon

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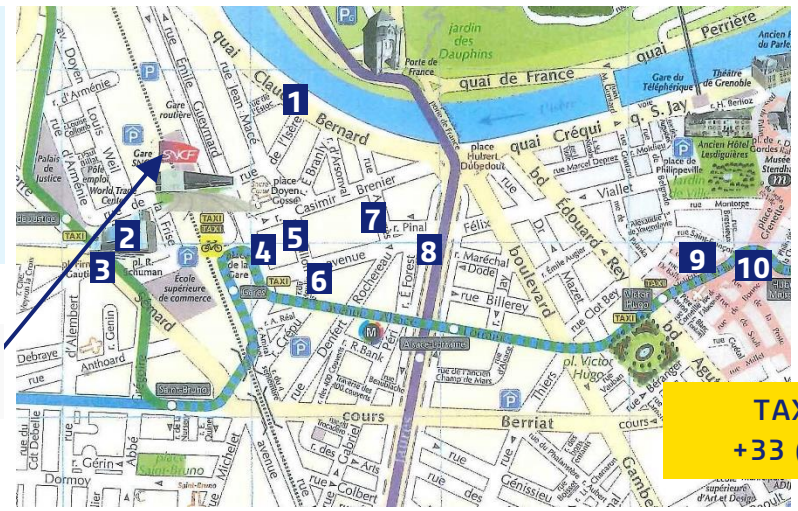

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For further information, please contact: Catherine Blanc, Agathe Chebre, Sonya Girodon
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List of Exhibitors

Exhibitors attending the 2023 ESRF User Meeting

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Kashiyama Vacuum Solutions

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XRnanotech

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Tutorials

6 February 2023

- Overall Programme

User Meeting 2023 - Tutorials
Monday 6 February 2023

	TUTORIAL TITLE	ORGANISERS	TIME	VENUE
T1	BAG Meeting	David Flot (ESRF) Montserrat Soler Lopez (ESRF) Max Nanao (ESRF)	**09:00 - 12:30**	**Auditorium**
T2	Getting the most from New in plate and plate to beam modalities on MASSIF-1	Serena Rocchio (EMBL) Nicolas Foos (EMBL) Matthew Bowler (EMBL) Didier Nurizzo (ESRF)	**09:00 - 12:00**	**ID30A-1**
			14:00 - 17:00	**ID30A-1**
T3	Volume image analysis	Alexander Rack (ESRF)	**09:00 - 17:00**	**MD-1-21**
T4	Science, communication and social media	Delphine Chenevier (ESRF)	**12:15 -13:15**	**Visitor Center**
T5	Data reduction for scattering experiments	Jérôme Kieffer (ESRF)	**09:00 - 12:00**	**500-501**
T6	Introduction to XAS	Kirill Lomachenko (ESRF)	**09:00 - 12:00**	**CIBB Seminar Room**
T7	EXAFS data analysis	Francesco D'Acapito (ESRF)	**14:00 - 17:00**	**337**
T8	Ab initio simulation of X-ray absorption spectroscopies using FDMNES	Yves Joly (ESRF)	**14:00 - 17:00**	**BEL-1-01**
T9	XPCS: X-ray Photon Correlation Spectroscopy	Federico Zontone (ESRF) Yuriy Chushkin (ESRF) Marco Cammarata (ESRF)	**09:00 - 12:00**	**EMBL 9-10**
T10	Coherent imaging analysis with PyNX: CDI (small angle and Bragg), ptychography, holotomography	Vincent Favre Nicolin (ESRF) Steven Leake (ESRF) Corentin Chatelier (CEA) David Simonne (Soleil)	**09:00 - 17:00**	**LOB-1-45**

Plenary Session

7 February 2023

- Programme
- Abstracts of lectures

Plenary Session
Tuesday 7 February 2023

MORNING SESSION		
09:00 - 09:05	Opening and welcome by the User Organisation	
09:05 - 09:50	**Invited Speaker** "*Order from disorder in sarcomeric Z-disks*" **Kristina Djinović-Carugo**, Head of EMBL Grenoble, France	Chair: **Adriana Miele** (UOC)
09:50 - 10:35	**Keynote Lecture 1** "*Synchrotron radiation enabled approaches to Human Bioarchaeology: Advances, problems and potential*" **Kirsi Lorentz**, The Cyprus Institute, Nicosia, Cyprus	Chair: **Barbara Fayard** (UOC)
10:35 - 11:00	*Break*	
11:00 - 12:30	**ESRF-EBS Facility and Director's Report** • Introduction - **Francesco Sette** • New Community Access Modes - **Annalisa Pastore** • New scientific opportunities - **Gema Martinez Criado** • Latest news from ID03 **Carsten Detlefs**, ID21 **Marine Cotte**, ID24 **Kirill Lomachenko** & questions for all	Chair: **Beatrice Ruta** (UOC)
12:30 - 14:00	*LUNCH break*	

AFTERNOON SESSION		
14:00 - 14:45	**Keynote Lecture 2** "*New frontiers and new tools in High Pressure Physics: an illustration with results on hydrogen rich systems*" **Paul Loubeyre**, CEA DIF, Université Paris-Saclay, France	Chair: **Guillaume Morard** (Chair of UOC)
14:45 - 15:30	**Keynote Lecture 3** "*The battery hub at ESRF: accelerating and standardizing battery characterization workflows*" **Sandrine Lyonnard**, CEA Grenoble, Laboratoire SyMMES, France	Chair: **Karsten Küpper** (UOC)
15:30 - 16:00	*Break*	
16:00 - 17:00	**Poster Clips**	Chair: **Alberto Martinelli** (UOC)
17:00 - 17:50	**Young Scientist Award 2023** President: **Cormac McGuiness**, ESUO President	Chair: **Guillermo Requena** (UOC)
17:50 - 18:00	Conclusion	
18:00 - 22:00	Poster Session, Poster Prize & Cocktail Dinner	

Order from disorder in sarcomeric Z-disks

K. Djinovic Carugo[1,2]

[1]European Molecular Biology Laboratory (EMBL) Grenoble, 71 Avenue des Martyrs, 38000 Grenoble, France;
[2]Department of Structural and Computational Biology, Max Perutz Labs, University of Vienna, Campus Vienna Biocenter 5, A-1030 Vienna, Austria **kristina.djinovic@embl.org**

The sarcomere is the minimal contractile unit in the cardiac and skeletal muscle, where actin and myosin filaments slide past each other to generate tension. This molecular machinery is supported by a subset of highly organised cytoskeletal proteins that fulfil architectural, mechanical and signalling functions. The ultra-structure of sarcomere is highly organized and delimited by Z-disks, which play a central role in the mechanical stabilization and force transmission.

In the Z-disks – the lateral boundaries of the sarcomere machinery – the protein α-actinin-2 cross-links antiparallel actin filaments from adjacent sarcomeres, and additionally serves as a binding platform for a number of other Z-disk proteins. In striated muscle cells, the Z-disk represents a highly organized three-dimensional assembly containing a large directory of proteins orchestrated in a multi-protein complex centred on its major component α-actinin, with still poorly understood hierarchy and three-dimensional interaction map. On the way to elucidate the molecular structural architecture of the Z-disk, the hierarchy of its assembly and structure-function relationships, we are studying binary and higher order sub-complexes of α-actinin using a combination of molecular biophysics, structural and biochemical approaches.

FATZ proteins interact with α-actinin and five other core Z-disk proteins, contributing to myofibril assembly and maintenance as a protein interaction hub. We determined the first structure and its cellular validation of α-actinin-2 in complex with a Z-disk partner, FATZ-1, which is best described as a conformational ensemble. We show that FATZ-1 forms a tight fuzzy complex with α-actinin-2 and propose a molecular interaction mechanism via main molecular recognition elements and secondary binding sites. The obtained integrative model reveals a polar architecture of the complex which, in combination with FATZ-1 multivalent scaffold function, might organise interaction partners and stabilise α-actinin-2 preferential orientation in the Z-disk. Finally, we uncover FATZ-1 ability to phase-separate and form biomolecular condensates with α-actinin-2, raising the intriguing question whether FATZ proteins can create an interaction hub for Z-disk proteins through membrane-less compartmentalization during myofibrillogenesis.

I will present our studies on the interaction of the major Z-disk protein α-actinin with FATZ and Z-portion of titin, forming dynamic fuzzy complexes, and discuss findings in view of asymmetric sorting of α-actinin and sarcomeric Z-disk architecture and assembly.

New frontiers and new tools in High Pressure Physics: an illustration with results on hydrogen rich systems

Paul Loubeyre[1]

[1]CEA/DIF/DPTA

High pressure physics is an old field which is advancing by the implementation of new tools. At least three facts can explain the great dynamism of the high pressure field. The frontier of exploration is now pushed up to the TPa (10 million bars), that is the pressure domain of planetary interiors. A different periodic table of atoms exists under a million bars and new materials can thus be synthesized, some with remarkable properties. Many measurements have been developed, particularly in front of synchrotrons, which enable a fine and detailed characterization of the properties of materials under extreme pressures almost as if at ambient pressure.

We will illustrate this renewal of the high pressure domain by focusing on some results around dense hydrogen systems. This theme is pushing the development of new experimental approaches and has implications for fundamental physics, materials science and astrophysics. In particular, we will discuss: hydrogen metallic phase; super-hydrides which are superconductors at high temperature; the superionic forms of water ice; the miscibility of H/He mixtures in the conditions of planetary interiors.

The battery hub at ESRF: accelerating and standardizing battery characterization workflows

Q. Jacquet[1], S. Tardif[2], J-F. Colin[3], C. Villevieille[4], L. Daniel[3], M. Chandesris[3], S. Lyonnard[1]

1. Univ. Grenoble Alpes, CEA, CNRS, IRIG-SyMMES, 38000 Grenoble, France
2. Department of Physics, Univ. Grenoble Alpes, CEA-IRIG, Grenoble, France
3. Department of Electricity and Hydrogen for Transport, Univ. Grenoble Alpes, CEA- LITEN, Grenoble, France
4. Univ. Grenoble Alpes, Univ. Savoie Mont Blanc, CNRS, Grenoble INP, LEPMI, 38000 Grenoble, France

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The development of high performance, safe and sustainable batteries for e.g. e-mobility, portable and stationary applications, demands overcoming major technological challenges. A more holistic approach is needed to accelerate the discovery of new materials and chemistries, which requires implementing advanced battery characterization in an integrated, automatized and unified manner. In fact, to accelerate our understanding of the variety of processes that dictate battery performance and ageing across an enormous range of relevant time and length-scales, we need to go beyond the traditional single-shot, sophisticated experiment and build centralized experimental frameworks based on multi-modal multi-techniques standardized data acquisition and analysis workflows [1].

To gather forces and efforts, and create impact beyond the usual competition rules and incremental progress, new mechanisms and methods organized into community-unified infrastructures are foreseen. In this spirit, we launched the pilot phase of the Grenoble Battery Hub, a new type of collaborative platform rallying the European battery community and the Large Scale Facilities around central scientific electrochemistry-related topics. This hub is designed to provide new access and cooperation modes, with the objective to accelerate Research & Innovation on batteries by setting an open scientific, technical and communication platform dedicated to promoting, carrying out and analysing cutting-edge neutron and X-ray investigations of battery components and devices. In this talk, we will describe the early stage of operation of the pilot phase started in sept. 2021, and present results obtained at ESRF using an array of techniques (diffraction, scattering, spectroscopy, imaging, tomography) to tackle an array of scientific questions (battery failure, ageing mechanisms, new chemistry reactions) with emphasis on cutting-edge correlative operando experiments conducted on standardized and/or smart batteries. We will discuss how the Hub organization shall go beyond standard collaboration schemes to tackle the challenges related to energy storage, and boost innovation by optimizing research workflow in relation to the European efforts and targets [2].

References

[1] - Advanced Energy Materials, 2022, 2102694. D. Atkins, […], S. Lyonnard*. Accelerating Battery Characterization Using Neutron and Synchrotron Techniques: Toward a Multi-Modal and Multi-Scale Standardized Experimental Workflow. DOI:10.1002/aenm.202102694

[2] - Advanced Energy Materials, 2022, 12, (17), 2102785. Amici, J., [...], Edström, K.*. A Roadmap for Transforming Research to Invent the Batteries of the Future Designed within the European Large Scale Research Initiative BATTERY 2030+. DOI:10.1002/aenm.202102785

User-Dedicated Microsymposium UDM 1

8 February 2023

Environmental sciences: challenges and opportunities under a new era of synchrotron light

UOC Organiser	Adriana Miele Beatrice Ruta
ESRF Organisers	Hiram Castillo Michel Jean-Louis Hazemann
Non-ESRF Organisers	Blanche Collin Alejandro Fernandez-Martinez Clément Levard Emmanuelle Montarges-Pelletier Géraldine Sarret

UDM1. Environmental sciences: challenges and opportunities under a new era of synchrotron light

Wednesday, 8 February 2023 - Microsymposium UDM1 IBS Seminar Room	
8:40 – 8:45	**Welcome from the organisers**

Morning session

	Session I	
8:45 – 9:25	**Keynote talk 1** High brightness and high coherence: what does it mean for imaging in environmental science?	**Chris Jacobsen** *Northwestern University, USA*
9:25 – 9:45	**User talk** *Nannoconus*: Analytical clue from past to future oceanic environments	**Rajkumar Chowdhury** *Université Grenoble Alpes, France*
9:45 – 10:05	**User talk** Porosity evolution during sustainable ironmaking with hydrogen probed by 4D synchrotron X-ray nano-tomography	**Yan Ma** *Max-Planck-Institut für Eisenforschung, Germany*
10:05 – 10:30	*Coffee break*	
	Session II	
10:30 – 11:10	**Keynote talk 2** Investigating the structure and reactivity of mineral–water interfaces using in situ high-resolution X-ray reflectivity	**Sang Soo Lee** *Argonne National Laboratory, USA*
11:10 – 11:30	**User talk** *In situ* measurements of interface evolution during mineral dissolution	**Bastien Wild** *Université Grenoble Alpes, France*
11:30 – 11:50	**User talk** Importance synchrotron X-ray radiation in determining technology critical elements speciation : application in (bio)hydrometallurgy	**Eric van Hullebusch** *Institut de Physique du Globe de Paris, France*
11:50 – 12:10	**User talk** Yttrium speciation in world-class phosphorite deposit	**Clément Bonnet** *Université de Montpellier, France*
12:10 – 13:30	*Lunch Break*	

UDM1. Environmental sciences: challenges and opportunities under a new era of synchrotron light

Afternoon session

	Session III	
13:30 – 14:10	**Keynote talk 3** Elucidating the uptake and transformation of nanophases by plant leaves	**Astrid Avellan** *CNRS, France*
14:10 – 14:30	**User talk** Arsenic speciation controls the immobilization echanism and stability of mixed valence iron minerals	**Jeffrey Paulo H. Perez** *GFZ German Research Centre for Geosciences, Germany*
14:30 – 14:50	**User talk** Spatially and temporally resolved mineral phase evolution and arsenic retention in microfluidic models of zerovalent iron-based water treatment	**Andreas Voegelin** *Swiss Federal Institute of Aquatic Science and Technology, Switzerland*
14:50 – 15:10	**User talk** Nanocrystalline manganese oxides: structure, reactivity, and their role in the geochemical cycle of trace elements	**Sylvain Grangeon** *BRGM, France*
15:10 – 15:35	*Coffee break*	
	Session IV	
15:35 – 15:55	**User talk** Carbonate-oxalate mineralogy from the Great Barrier Reef Under Synchrotron light	**Eduardo Villalobos-Portillo** *ESRF, France*
15:55 – 16:15	**User talk** From soil to bean: Unravelling the pathways of cadmium in cacao	**Hester Blommaert** *Université Grenoble Alpes, France*
16:15 – 16:35	**User talk** How soil organic carbon fractions and the phosphorus distribution at the microscale respond to land use change in Amazonian Dark Earth and Acrisol	**Luis Carlos Colocho Hurtarte** *ESRF, France*
16:35 – 16:55	**User talk** Rhenium speciation and reduction pathway in sulfidic settings	**Carolina Guida** *Université Grenoble Alpes, France*

High brightness and high coherence: what does it mean for imaging in environmental science?

Chris Jacobsen[1], Argonne Distinguished Fellow, Advanced Photon Source, Argonne National Laboratory, Lemont, IL, USA

[1] Professor, Department of Physics & Astronomy, Northwestern University, Evanston, IL, USA

We are fortunate to live in an era where quasi-time-continuous x-ray brightness and coherent flux have been increasing at incredible rate -- faster than Moore's law for integrated circuits! What opportunities does this create for imaging in environmental science? After a short reminder of what is meant by brightness, brilliance, and coherence, we consider the implications for imaging three aspects of natural materials: their morphology down to the nanometer scale, their crystallinity, and their elemental content (including chemical states). High coherent flux sources open new horizons for all of these experiments in the x-ray imaging zoo; however, George Orwell reminds us that "all animals are equal, but some animals are more equal than others." Thus some experiments are limited in the size of object that can be studied, while others are much less so, to the point where we can contemplate nanoscale imaging of centimeter-sized specimens. Radiation damage and specimen heating can limit what we can see, but with much variation depending on the type of sample, and the conditions we image it in. There may be a need for microscopes and detectors to catch up with the advancements in accelerators. Where will this lead? Dennis Gabor (who received the Nobel Prize for developing holography as a coherent imaging method) provided a nice perspective: "the future cannot be predicted, but futures can be invented."

Nannoconus: Analytical clue from past to future oceanic environments

R. Chowdhury[1], F. Giraud[1], A. Fernandez-Martinez[1], J. Cesar da-Silva[2], A. Kulow[2], R. Boudjehem[2], JL. Hazemann[2], J. Perez[3], B. Suchéras-Marx[4], G. Garbarino[5]

[1]Univ. Grenoble Alpes, Univ. Savoie Mont Blanc, CNRS, IRD, IFSTTAR, ISTerre, 38000 Grenoble, France, [2]Univ. Grenoble Alpes, CNRS, Grenoble INP*, Institut Néel, 38000 Grenoble, France, [3]SWING Beamline, Synchrotron SOLEIL, L'Orme des Merisiers, BP48, Saint-Aubin, Gif-sur-Yvette, F-91192, France, [4]Aix Marseille Univ, CNRS, IRD, INRAE, CEREGE, Aix-en-Provence, France, [5]ESRF, 71 avenue des Martyrs, 38043 Grenoble, France

The oceanic system, a huge part of the hydrosphere and the Earth, contains more than a billion micro-organisms in each liter of water. Some of them, such as calcifying unicellular photosynthetic algae, have significant influence on the global carbon cycle. Apart from this, they are crucial for deciphering major changes in environmental oceanic conditions recorded in their calcified exoskeleton. These calcifying unicellular photosynthetic algae are particularly well-represented in the fossil marine record, as nannofossils (remains of their exoskeleton) from ~ 210 million years. Thus, both physical and chemical characterization of such individual nannofossil will help to understand the factors controlling the past environmental oceanic conditions. This information can give keys for understanding present and future perturbations in the oceanic system.

Here, we present an experiment on a set of extinct algal nannofossils (micrometric size), *Nannoconus*, the main photosynthetic bio-carbonate producers in 150 million years old oceans. The calcareous exoskeleton (5-30 μm) produced by the *Nannoconus* remained preserved in oceanic carbonate sediments. It is characterized by a conical shape with successive arrangement of calcitic plates (~100 nm in thickness) spanned around a central canal. Based on the differential shape, size and arrangement of the plates of the exoskeleton, there are 9 recognized morpho-groups that thrived in a time span of 50 million years. The detailed physical and chemical study of this skeletal calcite was non-existent to assign a link between the successful calcification and the changes in ocean chemistry. To better understand the link between the physical and chemical characterization of the *Nannoconus* and the changes in ocean conditions, we used the X-ray ptychographic computed tomography at the SWING beamline in SOLEIL, the French Synchrotron (June 2022). The experiment used nearly 1000 tomographic projections between 0° and 180° with a beam size of 5 μm and a step size of 1.5 μm for each ptychographic scan. This resulted in a very fine spatial resolution (~50-60 nm), useful for the quantitative 3D volume reconstruction and density analysis of the shell. The ongoing treatment of the results yielded the values of the electron density of the specimen, which can be used to obtain individual mass. This will help to decipher directly the composition the exoskeleton to nanometric variation and possible evolution of the size and weight over time. The direct and precise values of the mass will quantitatively add a value to what makes *Nannoconus*, an important pelagic bio-carbonate producers.

Chemical analyses of the exoskeletal calcite of the *Nannoconus*, are planned at ID21 in the next semester. They will qualitatively indicate the paleo-environmental conditions.The mentioned time interval (~ 150-100 Million years) is indeed characterized by some major environmental perturbations i.e. in the global carbon cycle, and in the dissolve oxygen in seawater. Data from all these experiments are expected to give some clues concerning both the response and the adaptation of *Nannoconus* to the changing environmental conditions in the oceanic system.

Porosity evolution during sustainable ironmaking with hydrogen probed by 4D synchrotron X-ray nano-tomography

Yan Ma[1], Yen-Fan Wang[1], Katrin Bugelnig[2], Julie Villanova[3], Guillermo Requena[2,4], Dierk Raabe[1]

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[2] Institute of Materials Research, German Aerospace Center (DLR), Linder Höhe, 51147 Cologne, Germany
[3] ESRF-The European Synchrotron, 71, Avenue des Martyrs, CS 40220, 38043 Grenoble Cedex 9, France
[4] Lehr- und Forschungsgebiet Metallische Strukturen und Werkstoffsysteme für die Luft- und Raumfahrt, RWTH Aachen University, 52062 Aachen, Germany

Iron- and steelmaking cause more than 7% global CO_2 emissions due to deploying carbon-based substances (*e.g.*, coal and coke) as reductants for iron ores, making it a key driver of global warming [1]. Fossil-fuel free ironmaking is indispensable for reducing these massive anthropogenic CO_2 emissions. Hydrogen-based direct reduction (HyDR) is one of the most promising solutions to sustainable ironmaking. HyRD by nature is a multistep solid-gas reaction, involving several complex phenomena, such as non-volume conserving phase transformations, mass loss and transport, *etc*. [2,3] All these can lead to pore formation. However, a 2D post-mortem microstructural analysis is unsatisfactory to reveal the actual porosity, especially the connectivity of its networks. Yet, this information is crucial to better understand its role in the outbound mass transport of reduction product. In this study, we employed 4D synchrotron X-ray nano-tomography to characterize the porosity during HyDR of hematite. The time-resolved tomography scans showed the formation and evolution of the pores and their percolation features. The pore formation mechanism and the correlation between porosity evolution and reduction kinetics during HyDR are discussed.

References

[1] - D. Raabe, C. C. Tasan, E. A. Olivetti, Nature **575**, 64 (2019)
[2] - S. H. Kim, X. Zhang, Y. Ma, *et al.*, Acta Materialia **212**, 116933 (2021)
[3] - Y. Ma, I. R. Souza Filho, Y. Bai, *et al.*, Scripta Materialia **213**, 114571 (2022)

Investigating the structure and reactivity of mineral–water interfaces using in situ high-resolution X-ray reflectivity

Sang Soo Lee[1]

[1]Chemical Sciences and Engineering Division, Argonne National Laboratory

Adsorption and desorption at mineral–water interfaces control the mobility and bioavailability of nutrients and toxic elements in natural environments. Understanding these interfacial processes can provide a direct insight into the reaction mechanisms. Obtaining this fundamental knowledge can largely benefit from our ability to observe adsorbed ion distributions at atomic level. In this presentation, I show how ion charge and hydration influence the speciation and dynamics of metal ions adsorbed at mineral–water interfaces based on in-situ observations using synchrotron-based high-resolution X-ray reflectivity [1,2]. I also highlight how these structures can be modified with varying solution composition and external environmental parameters [3]. These experimental observations are compared with computational simulations to provide a more detailed insight into molecular-scale behaviours in complex interfacial phenomena [3,4]. Finally, I discuss new opportunities and challenges for studies of the interfacial geochemistry using the high brilliance and coherence from the 4th generation source of X-rays.

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In situ measurements of interface evolution during mineral dissolution

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The dissolution of rock-forming minerals is relevant to a broad range of topics in the area of geosciences and environmental sciences. The weathering of silicates controls atmospheric CO_2 over geological timescales and this process is currently being harnessed in the framework of carbon capture and storage strategies. The dissolution of minerals also controls the long-term production of inorganic nutrients and the release of metal contaminants at mining sites. Dissolution rates and associated fluxes are however difficult to predict, partly due to our incomplete understanding of the evolution of interfaces during dissolution. In particular, under certain conditions, the formation of Surface Alteration Layers (SALs) formed by interface-coupled dissolution-reprecipitation at fluid/mineral interfaces may constitute a diffusion barrier impacting the overall dissolution process.

A series of studies are outlined here, that aim at relating the properties of SALs to the dissolution rate of the underlying mineral. The textural and transport properties of SALs were probed *in situ* by combining Grazing-Incidence Small-Angle X-ray Scattering (GISAXS) and X-Ray Reflectivity (XRR) measurements. The apparent diffusivity of SALs was determined by following the evolution of scattering length density profiles during tracer percolation experiments, while the atomic structure of these layers and the exact nature of the transformation associated with their formation was probed using *in situ* X-ray Pair Distribution Function analysis (PDF).

Combined with the results of other *in situ* surface-sensitive techniques currently under development, these results shed light on molecular-scale processes underlying mineral dissolution rates observed at the macroscopic scale.

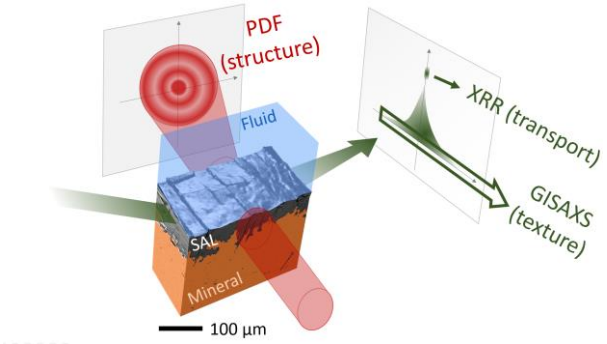

Figure 1: Overview of the approach used in this study

Importance synchrotron X-ray radiation in determining technology critical elements speciation : application in (bio)hydrometallurgy

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The energy transition will shift the demand from fossil fuels to technology critical elements (TCEs) needed for the growing production of electric vehicles and their batteries, wind turbines, solar panels and kilometres of electricity grids. Consequently, by 2050, more metals will have to be extracted from the subsoil than have been extracted and consumed by mankind since its inception, and the waste associated with this industry will have to be managed: this can only be done if the associated risks and environmental impacts are controlled and the life cycle of these elements is optimised by minimising the outflow of the cycle and maximising recycling and recovery, while reducing the consumption of our energy resources [1].

In addition to the huge amount of primary mining wastes produced each year, metallurgical refining and recycling of urban waste generates increasing volumes of residues containing sometimes even higher levels of TCEs compared to primary ores. These TCEs are encountered in complex mineralogical combinations in urban wastes such as e-wastes [2, 3], municipal solid waste incineration ashes, slags and sludge from metallurgy [4] and chemical industry residues such as spent catalysts.

However, TCEs recovery from secondary resources can be a challenge. The variable TCEs content and complex mineralogical occurrence together with various materials often render conventional extraction processes such as pyrometallurgy unsuitable. Even if classical extraction methods applied to high-grade ores are often inefficient, in contrast (bio)hydrometallurgical extraction processes can be more suitable TCEs recovery methods for complex secondary resources.

The elemental and mineralogical characterisation of secondary resources has been reported to be of high importance for unravelling the leaching and recovery mechanisms while optimizing the (bio)processes efficiency [4, 5]. X-ray Absorption Near Edge Spectroscopy (XANES) capable of detecting changes in oxidation state while Extended X-ray absorption fine structure (EXAFS) may provide information on the chemical environment and bond distances at macro- or micro-scales. Depending on the required particle size, other solid state characterisation techniques can be employed, including micro-fluorescence spectroscopy (μ-XRF) and X-ray diffraction powered by a high-energy X-ray source from a synchrotron radiation facility.

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Yttrium speciation in world-class phosphorite deposit

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Phosphorite rocks are mined for the production of P fertilizers and contain rare earth elements (REEs, that include lanthanides and yttrium from ~200 to 20000 ppm) [1]. The latter are considered as critical metals essential for green technologies (magnets, catalysts, batteries) and their demand is growing exponentially. Consequently, phosphorite deposits are considered as an unconventional resource of REE, where co-valorization techniques are increasingly being considered [2]. This opportunity allows the sustainable and clean development of phosphate resources as well as involving it in the energy transition. Understanding the crystal chemistry of REEs in phosphorites is therefore a crucial aspect to decipher for the further development of effective co-recovery techniques.

Thus, in order to make a robust contribution to this issue, we used the ESRF synchrotron radiation which benefits from the EBS upgrade at the BM23 beamline, to perform X-ray absorption at the yttrium *K*-edge on REE-diluted rock samples from the largest world-class phosphorite deposit in Morocco.

Our study demonstrates that REEs are mainly hosted by the fluorapatite mineral. We also propose that their binding can be either i) a mixture of ~60% substituted in the Ca(2)-site and ~40% adsorbed as an inner shell complex at the c-axis channel or, ii) a substitution in the Ca(2) site in a carbonated fluorapatite (francolite). Further *in-situ* µXAS analysis will determine which of the two assumptions is more appropriate. In both instances, our findings suggest that REEs are more recoverable than expected, either as an adsorbed fraction that can be efficiently desorbed, or as hosted in the easily leachable francolite mineral phase.

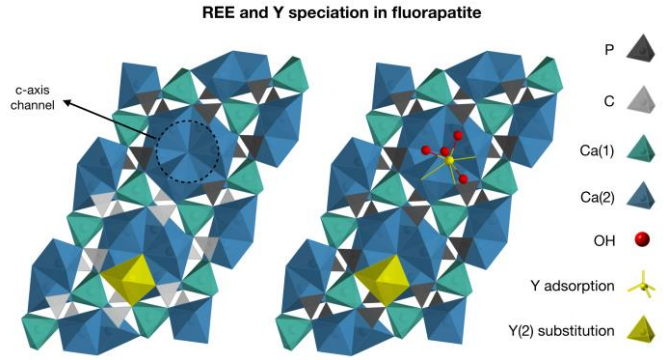

Figure 1: Fluorapatite crystal structure showing potential Y speciation models, a) mixture of ~60% substituted in the Ca(2) site and ~40% adsorbed as an inner-shell complex, b) substituted in the Ca(2) site in a francolite

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Elucidating the uptake and transformation of nanophases by plant leaves

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The delivery of (micro)nutrients to crop plants through their leaves is an interesting strategy to decrease soil contamination and reduce agricultural inputs. Until now, foliar fertilization has not been widely used due to the low bioavailability of salts when deposited on leaves. In contrast, nanophases with the right physical-chemical properties can not only adhere to plant surfaces better, but they also can penetrate the plant leaf and reach various plant compartments. While nanophases could thus be an interesting tool to reduce the environmental impact of agricultural practices through a reduced wash-off, the fate of these particles after their foliar deposition still need to be elucidated.

Our research aims to elucidate the mechanisms responsible for the uptake, transformation and mobility of nanophases in plants after their deposition on leaves. To study the journey of these nanophases, one need to address their cellular localization and associated transformations in hydrated tissues. In this presentation, we will discuss examples demonstrating how the use of synchrotron radiation and spatially resolved techniques has helped us to unravel these nanophase-plant interactions.

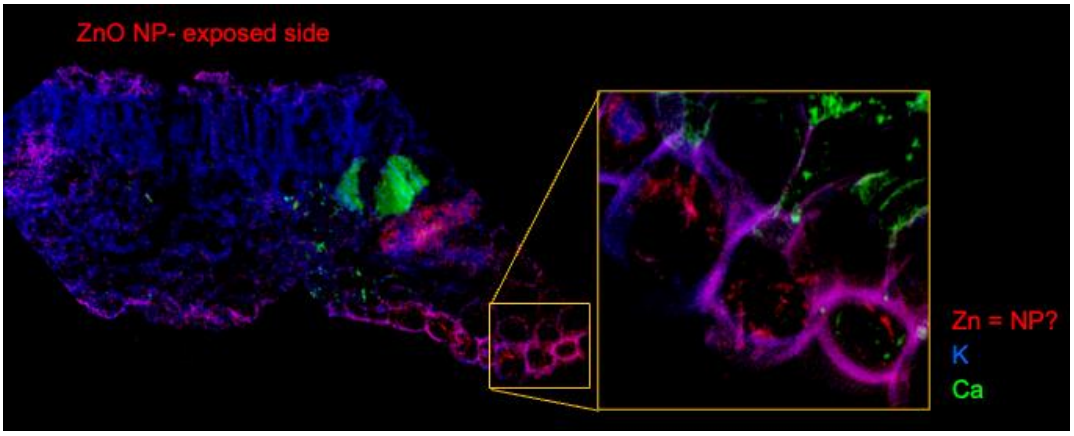

Figure 1: X-ray fluorescence map collected on ID-21 at ESRF on hydrated, frozen cross-section of a pepper leaf one month after leaf deposition of ZnO NPs.

Arsenic speciation controls the immobilization mechanism and stability of mixed valence iron minerals

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Green rust is a mixed valence layered iron mineral that often forms in ferruginous (i.e. Fe^{2+}-rich and oxygen-poor) environments. Due to its nanoparticulate nature and redox reactivity, it can influence the availability of nutrients and trace element, and the mobility of many contaminants (e.g., As, Cr) [1]. Among common iron minerals, green rust has one of the highest arsenic uptake capacities at circum-neutral pH conditions [2]. However, the mechanism and interdependencies between green rust and arsenic species, and their role in formation and transformation reactions in the subsurface, are still poorly understood. Combining high-resolution electron microscopy (S/TEM) and synchrotron-based X-ray spectroscopy and scattering techniques (XAS/PDF), we have shown that the oxidation state of arsenic highly affects its interaction with mixed valence iron minerals, and green rust in particular. Specifically, (i) As removal efficiency, (ii) As binding mechanism to green rust [3], (iii) green rust formation and transformation kinetics and (iv) the long-term stability of As-bearing iron minerals are all affected by As speciation [4]. Overall, our results provide new insights on iron mineral-metal interactions in anoxic environments, as well as its potential for mineral-based technologies for groundwater remediation.

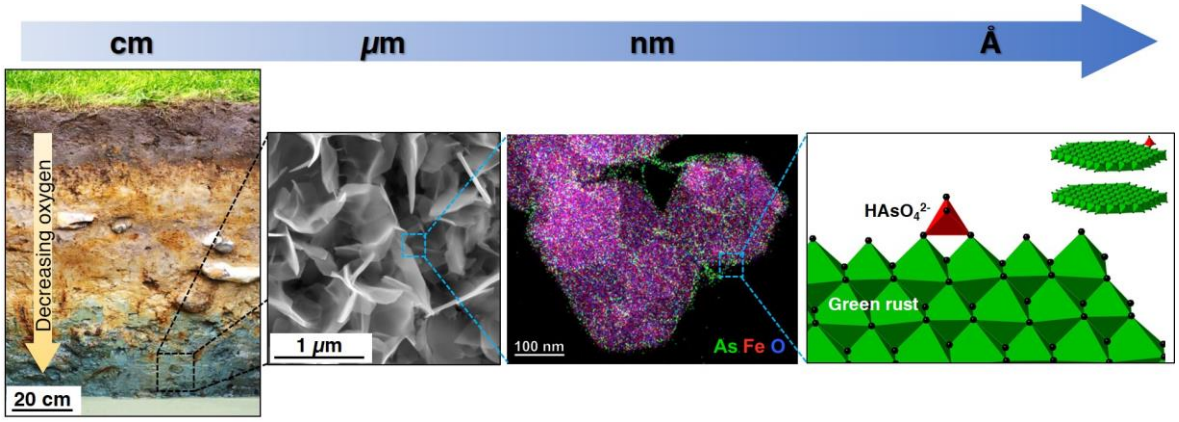

Figure 1: Investigation of green rust and arsenic species from micro- down to molecular scales using a combination of high resolution electron microscopy and synchrotron-based X-ray techniques.

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Spatially and temporally resolved mineral phase evolution and arsenic retention in microfluidic models of zerovalent iron-based water treatment

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Arsenic (As) is a toxic element, and elevated levels of geogenic As in drinking water pose a threat to the health of several hundred million people worldwide. In this study, we used microfluidics in combination with optical microscopy and spatially resolved X-ray spectroscopy to investigate zerovalent iron (ZVI) corrosion, secondary iron (Fe) phase formation, and As retention processes at the pore scale in ZVI-based water treatment filters.

Two 250 µm thick microchannels, each filled with a single layer of quartz and ZVI grains were operated intermittently (12 h flow/12 h no-flow) with synthetic groundwater over 13 and 49 days. During operation, we followed the corrosion of ZVI and the formation and transformation of Fe mineral phases with optical microscopy. After operation, the microchannels were resin-embedded for analysis by synchrotron-based micro-focused X-ray fluorescence spectrometry (µ-XRF), X-ray absorption spectroscopy (µ-XAS), chemical imaging and full-field XAS to gain insights into the spatial distribution of Fe, As and other elements, the distribution of Fe mineral phases, and the redox speciation of As.

Optical microscopy-based time-lapse movies provide novel insights into the highly dynamic mineral phase evolution in the filter pore space during operation. In combination with the synchrotron data collected after operation, the results show that intermittent filter operation leads to cyclic phase transformations between green rust and lepidocrocite and gradual formation of magnetite close to ZVI grains as longer-term host for As. The results also show that upstream P removal leads to downstream Ca-carbonate precipitation, which in turn can promote anoxic ZVI corrosion. The study thus highlights the complex spatiotemporal coupling of various geochemical processes in the pore space. The results from this work are not only of interest with respect to the optimization of ZVI-based drinking water treatment, but also with respect to the use of ZVI in groundwater remediation.

Methodologically, this work shows that spatially and temporally resolved studies in micromodels can offer unprecedented insights into geochemical processes at the pore scale under conditions of kinetic and transport limitations. In the future, further advances in the study of geochemical processes at the pore scale can be achieved by combining micromodel experiments with in-situ spatially resolved synchrotron X-ray spectroscopies.

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Nanocrystalline manganese oxides: structure, reactivity, and their role in the geochemical cycle of trace elements

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"Nanocrystalline manganese (oxy)hydroxides" (often referred to as "Mn oxides") is a generic term for a large family of minerals having layered, tunnel, or compact structures [1]. Amongst them, layered structures, and more particularly vernadite (δ-MnO_2; the nanocrystalline and disordered variation of birnessite), are certainly the most reactive form. Vernadite reactivity stems from its minute size (typically 10 nm in the layer plane and 2-3 nm perpendicular) and from its large layer charge, which is about 3 times higher than that of smectite [2]. As a result of this high reactivity, vernadite controls or influences the fate of several nutrients, metals, metalloids and actinides in oxic soils such as brown soils, in the water column of freshwaters, and in sediments, including sea floors. In addition, due to the potential coexistence in its structure of Mn^{2+}, Mn^{3+}, and Mn^{4+}, vernadite is redox active, and can oxidize a large number of organic molecules, including pollutants [3].

However, vernadite is observed to be unstable under certain environmental conditions and under certain structural variation. When unstable, vernadite tends to transform to tunnel structures with time, following a solid-state recrystallization and growth mechanism [4]. Upon transformation, chemical elements such as metals, initially adsorbed at vernadite surface, are susceptible of either being released in solution or incorporated in the resulting solid, with two antagonist effects about the mobility of the element that was initially adsorbed.

Here, we will review the current state-of-the-art knowledge of vernadite crystal structure, of its evolution with time, and its impact on the mobility of both naturally occurring soil chemical elements and pollutants. In this view, the kinetics of trace elements uptake by vernadite and of solid-state transformation, which are particularly important to the understanding of uptake mechanisms in the critical zone, will be discussed. The long-term fate of vernadite, under slowly fluctuating redox conditions, will also be discussed, with the example of polymetallic nodules that are found on sea floors.

Finally, the structural similarities between vernadite, clay minerals, and fougèrite (a layered mineral that is found for example in reductisols, where it controls the fate of anionic pollutants and nutrients [5]) will be discussed. The twofold aim will be to discuss how the experimental methodologies developed for a given mineral (e.g., synchrotron in-situ XRD, PDF, XAS, and X-ray computed diffraction tomography) can be transposed to an another, and how the combined study of the different soil layered minerals may provide insights into the geochemical cycles of elements, including pollutants.

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Carbonate-oxalate mineralogy from the Great Barrier Reef Under Synchrotron light

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The Great Barrier Reef is the biggest and most complex reef ecosystem in the world. It represents 10 percent of the world's coral reef ecosystems by area. Due to warmer seas leading to coral bleaching and the impact of human pollution, more than half of the coral has been lost. Understanding the changes in structure of the corals during the bleaching process is essential to find solutions against it. This talk shows the first results from this study performed at the ID21 beamline of the ESRF [1].

This study is focused on the correlation between the mineralogical, geochemical and microbial characteristics of sediments and (mineralised) corals from the Low Isles reef near Port Douglas. By characterizing the minerals present in the corals, we show how susceptible certain corals are to acidification form the rising sea temperatures. Some oxalates such as the rare mineral weddellite ($Ca(C_2O_4)\cdot(2.5-x)H_2O$) are less resilient than aragonite or high-Mg calcite. Associating the different phases in the Ca–C–O system with the levels of weathering is crucial to the understanding future damage. µXRF synchrotron mapping helped us to identify the distribution and concentration of critical elements (from Si to Ca) which were used as target zones to see differences between the Ca K-edge µXANES signals. Differences between phosphorus and potassium hot-spots from bleached and non-bleached corals shows that contamination from fertilizer run-off from land-based agriculture results in changes to the trace element signatures in coral.

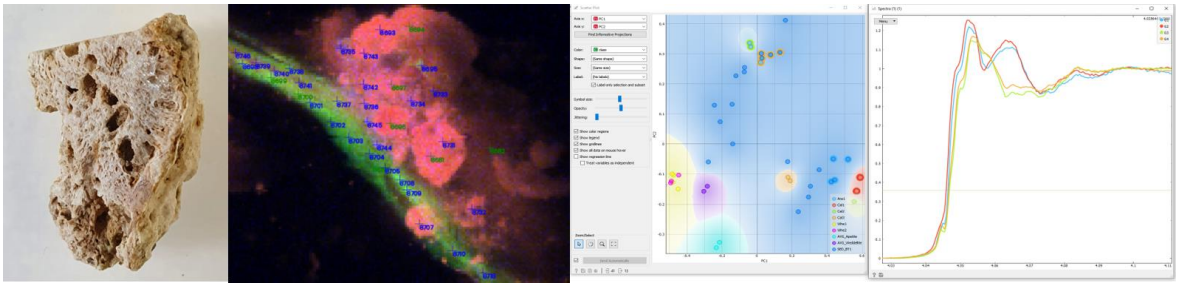

Figure 1: Workflow from sample to data of the Ca Coral experiment at ID21.

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From soil to bean: Unravelling the pathways of cadmium in cacao

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The accumulation of the potentially toxic metal cadmium (Cd) in cacao beans has recently become a subject of intense research after the European Union and the Codex Alimentarius lowered its legal limits in chocolate. The research on strategies to reduce cadmium (Cd) accumulation in cacao beans is currently limited by a lack of understanding of the Cd transfer pathways within the cacao tree 1. The study of samples at environmental Cd concentrations is challenging because of the low Cd content (a few mg kg-1 dw). Here, we studied the transfer of Cd from soil to the nib in a high Cd accumulating cacao cultivar by analyzing total elemental concentrations, Cd stable isotope fractionation, Cd speciation and localization. The plant Cd concentrations were 10-28 higher than the topsoil Cd concentrations. The largest fraction (57%) of total plant Cd was present in the branches where it was primarily bound to carboxyl-ligands (60-100%) and mainly localized in the phloem rays and phelloderm of the bark. Cadmium in the nibs was mainly bound to oxygen ligands (60-90%), with phytate as the most plausible ligand. A scheme of Cd pathway from roots to nib was proposed, and compared with other species. This study extended the limited knowledge on Cd accumulation in perennial, woody crops and revealed that the Cd pathway is markedly different than in annual crops, which has implications for mitigation strategies 2.

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How soil organic carbon fractions and the phosphorus distribution at the microscale respond to land use change in Amazonian Dark Earth and Acrisol

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The conversion of tropical forest for cassava cultivation is known to decrease soil organic carbon (OC) and nutrient contents of highly weathered soils in the tropics. Amazonian Dark Earth (ADE) may be resistant to this degradation because of their historical anthropogenic amelioration leading to higher soil OC and P concentrations. In this study, we assessed the effect of land use change on OC dynamics under tropical conditions and how this is related with P distribution at the microscale. We analyzed ADE and an adjacent Acrisol from Manaus (Brazil), both under forest and cassava plantation. The land use change induced a decrease of OC related to both particulate and mineral-associated organic carbon. Simultaneously the P content decreased by approximately 80% by land use change, whereas the relative proportion of organic P increased. This indicates a legacy effect of anthropogenic amelioration in the ADE for P but not for OC. Land use change tightened the OC-P relations in the mineral-associated OC fractions, which was also reflected at the microscale. Using NanoSIMS we found µm-sized P hotspots that were more co-localized with OC-dominated areas by land use change. Correlative measurements with synchotron-based µ-XRF and µ-XANES demonstrate a high spatial heterogeneity of different P species. A short-term incubation experiment with ^{13}C glucose showed a delayed and lower respiration through land use change. In our contribution, we will discuss distinct C and P interactions in microscale compartments and how these respond to land use change in highly weathered tropical soils.

Rhenium speciation and reduction pathway in sulfidic settings

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The geochemical behavior of redox-sensitive trace metal rhenium (Re) has been postulated to record information about the extent of low oxygen depositional conditions in the oceans. In the modern oxygenated ocean, Re speciation is dominated by the geochemically inert perrhenate $Re(VII)O_4^-$ anion at concentrations of ~40 pM [1]. Within oxygenated (pore)water, Re monovalent oxyanion does not react directly with Al/Fe/Mn oxyhydroxides, clay materials, or particulate humic substances [2,3]. The highest levels of Re are found in sulfide-rich euxinic settings, where concentrations of hundreds of ppb give this trace element the highest level of enrichment found in black shales [4]. Indeed, in the presence of sulfide, the oxyanion $Re(VII)O_4^-$ undo transformation via substitution of the oxyanion O atoms by S atoms, forming thioperrhenate species ($Re(VII)O_{4-x}S_x^-$) in a similar way to Mo [5]. Understanding this reaction is further required to extract and/or recycle Rhenium, a rare critical metal fundamental in high-tech products and emerging innovations industries, with limited possible substitution [6]. This element is mainly used in high-temperature superalloys to manufacture turbine blades for jet engines, in power generation applications, and platinum-rhenium catalysts in the petrochemical industry.

Our study aims to identify and follow for the first time with XAS spectroscopy the thioperrhenate species during Re thiolation and, subsequently, the steps leading to the formation of $Re^{IV}S_2(S)$ or $Re_2S_7(s)$ particles. This study aims to shed new light on the regulation of Re deposition in sulfide environments, to contribute to the development of improved mining and recycling methods, and to open the way for the use of Re as a chemical reporter.

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User-Dedicated Microsymposium UDM 2

8 February 2023

Tomography at BM18

UOC Organiser	Barbara Fayard Guillermo Requena
ESRF Organisers	Vincent Fernandez Paul Tafforeau
Non-ESRF Organiser	Camille Berruyer Joseph Brunet Peter Lee

- Programme
- Abstracts of Keynote & User Talks

UDM2. Tomography at BM18

Wednesday, 8 February 2023 - Microsymposium UDM2 ESRF MD-1-21		
9:00 – 9:15	**Welcome and Introduction** Present and future state of BM18	**Paul Tafforeau** *ESRF, France*

Morning session

	Session I	
9:15 – 9:45	**Keynote talk 1** Creating Gold Standard, Ground-truth Dataset for the Human Organ Atlas from BM18	**Claire Walsh** *University College London, UK*
9:45 – 10:00	**User talk** The fatal trajectory of pulmonary COVID-19 is driven by lobular ischemia and fibrotic remodeling *Zoom Session*	**Max Ackermann** *Helios University Clinics Wuppertal, University Mainz, Germany*
10:00 – 10:15	**User talk** Contribution of hierarchical phase-contrast tomography to the multi-scale anatomical study of human organs: example of the heart and spleen	**Alexandre Bellier** *CHU Grenoble Alpes, France*
10:15 – 10:30	**User talk** Cinematic Visualization: From Organs to Cells and Physiology	**Klaus Engel** *Siemens Healthineers, Erlangen, Germany*
10:30 – 11:00	*Coffee break*	
	Session II	
11:00 – 11:30	**Keynote talk 2** High-energy phase contrast micro computed tomography on BM18: A progress report	**Simon Zabler** *Fraunhofer Institute IIS, Germany*
11:30 – 11:45	**User talk** Addressing industrial challenges in tomography using BM18 beamline	**Sofiane Terzi** *Novitom Grenoble, France*
11:45 – 12:00	**User talk** Opportunities for Multi-Scale Imaging in Advanced Batteries	**Paul Shearing** *University College London, UK*
12:00 – 12:15	**User talk** Performance and Transmitter-Receiver Trade-offs in Giant Frog Acoustic Communication	**Morgane Sowinski** *Muséum National d'Histoire Naturelle, Paris, France*
12:15 – 12:30	**User talk** Using BM18 for multi-scale tomography of large fossils: the pelvic region of the near-tetrapod *Elpistostege*	**Per Ahlberg** *Uppsala University, Sweden*
12:30 – 14:00	*Lunch Break*	

UDM2. Tomography at BM18

Afternoon session

	Session III	
14:00 – 14:30	**Keynote talk 3** How synchrotron scanning is changing the evolutionary origins of mammals	**Julien Benoit** *University of the Witwatersrand, South Africa*
14:30 – 14:45	**User talk** GEOSCIENCES AT BM18: Earthquakes, Volcanoes and CO_2 storage	**Benoit Cordonnier** *ESRF/University of Oslo*
14:45 – 15:00	**User talk** Correlative imaging for additive manufacturing of metallic materials	**Pierre Lhuissier** *Simap Grenoble INP, France*
15:00 – 16:00	**Discussion**	
16:00	**Closing remarks**	

Creating Gold Standard, Ground-truth Dataset for the Human Organ Atlas from BM18

Claire Walsh[1], Hannah Coleman[1], Joseph Brunet[1,2] , Shahrokh Rhamani[1], Camille Berruyer[1,2], , Max Ackermann[3], Danny D Jonikg[4], Christopher Werlein[5], Alexandre Bellier[6], Simon Walker-Samuel[1], Joseph Jacobs[1], Paul Tafforeau[3] and Peter D Lee[1]

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HiP-CT on BM18 enables acquisition of large 3D multi-scale data sets of human organs in increasingly shorter times [1]. These datasets have great potential utility to the physiology, clinical imaging fields, but key to unlocking the potential are generalisable machine learning (ML) approaches to automate image segmentation. A major obstacle to developing these ML algorithms is the lack of sufficient high-quality ground-truth data [2].

Gold Standard or ground-truth segmentations should ideally be produced through segmentations and proof-reading stages by multiple experts. There are several challenges to this: i) As HiP-CT is a new technique there remain a shortage of experts; ii) HiP-CT data sets are large and the experts do not necessarily have access to the computational infrastructure needed to perform the segmentations. iii) Segmentations are laborious limiting the amount of time a single annotator can spend on the task before performance drops.

We present the current process of creating HiP-CT gold-standard segmentations, the challenges still faced and the efficacy of each method in terms of the dataset size created, the success in its use as a training dataset for machine learning and the time it took to create the dataset.

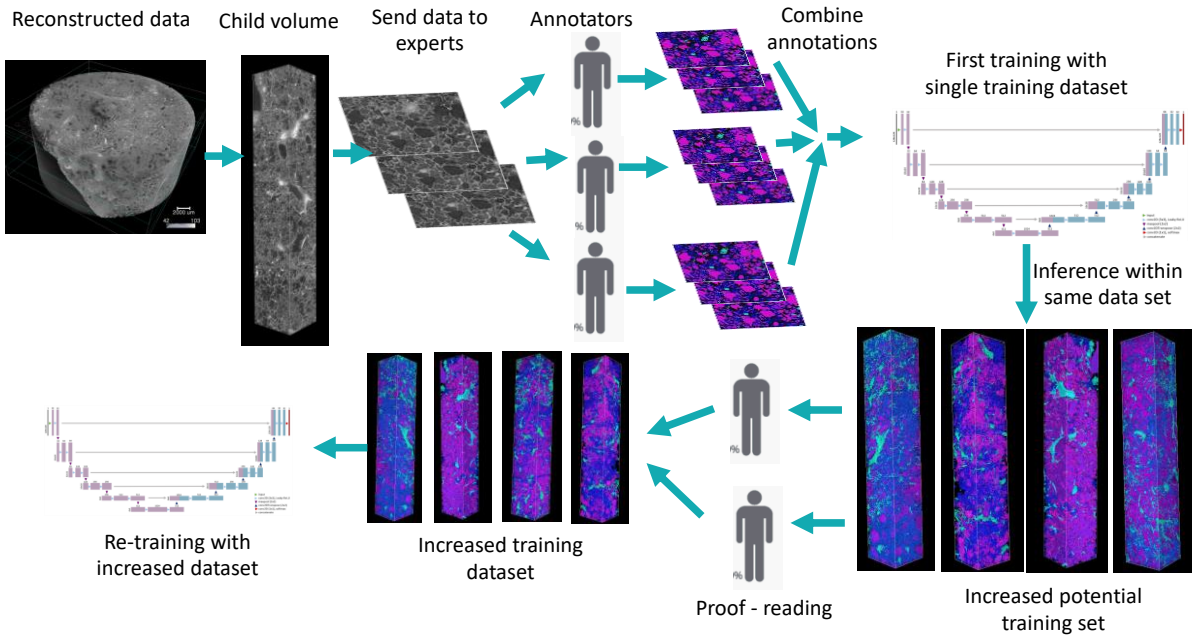

Figure 1: The pipeline for the creation of gold standard training data

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The fatal trajectory of pulmonary COVID-19 isdriven by lobular ischemia and fibrotic remodelling

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COVID-19 pneumonia revealed perivascular inflammation, an endothelial injury, microangiopathy, and an aberrant blood vessel neoformation by intussusceptive angiogenesis. The pathomechanism by which SARS-CoV2 causes the fatal trajectory of pulmonary pathology COVID-19 still remains vague. We studied a total of 85 lungs (COVID autopsy ; influenza A; ILD explants; healthy controls) using the highest resolution Synchrotron radiation-based hierarchical phase-contrast tomography, scanning electron microscopy of microvascular corrosion casts, IHC, MALDI-TOF, and analysis of mRNA expression and biological pathways. Plasma samples from all disease groups were used for liquid biomarker determination using ELISA. The observed mosaic appearance of COVID-19 in conventional lung imaging resulted from microvascular occlusion and secondary lobular ischemia. The length of hospitalization was associated with increased intussusceptive angiogenesis. This was associated with enhanced angiogenic, and fibrotic gene expression demonstrated by molecular profiling and metabolomics. Plasma analysis confirmed distinct fibrosis biomarkers (TSP2, GDF15, IGFBP7, Pro-C3) that predicted the fatal trajectory in COVID-19. Pulmonary severe COVID-19 is a consequence of secondary lobular microischemia and fibrotic remodelling, resulting in a distinctive form of fibrotic interstitial lung disease that contributes to long-COVID.

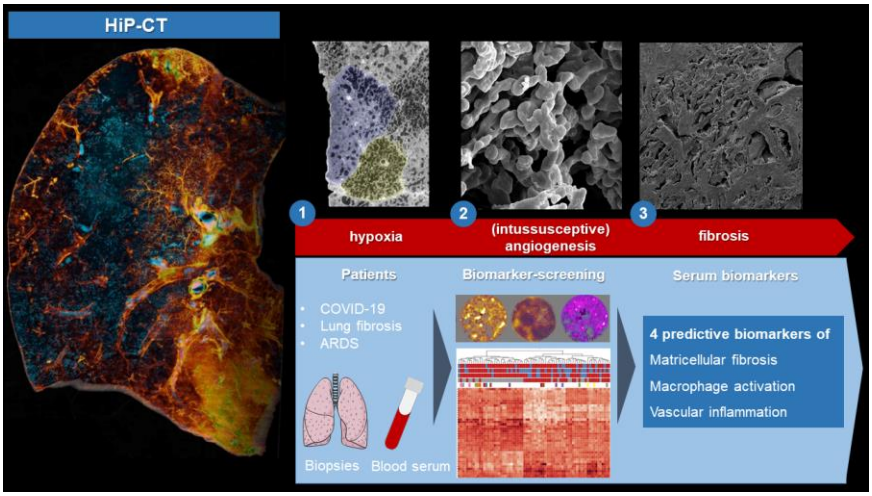

Figure 1: With the help of synchrotron-based hierarchical phase-contrast tomography (HiP-CT), a mosaic-like hypoxic undersupply of the smallest functional unit of the in severe COVID-19 lungs, the lung lobules, could be shown for the first time. This hypoxia and vascular damage caused by the SARS-CoV-2 virus leads to excessive formation of new blood vessels, so-called intussusceptive angiogenesis, which leads to scarring and fibrosis of the lung tissue in a very short time via inflammatory processes. In order to identify potential therapeutic targets or progression biomarkers, blood serum and biopsy tissue from patients with different COVID-19 progressions, pulmonary fibrosis (IPF) and acute lung injury (ARDS) were analysed and validated in a broad screening approach using proteomics and metabolomics. Three matricellular biomarkers and one macrophage-derived biomarker were identified as predictive blood-biomarkers that predict the progression of the scarring process.

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Contribution of hierarchical phase-contrast tomography to the multi-scale anatomical study of human organs: example of the heart and spleen

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[5] Centre for Advanced Biomedical Imaging, University College London, UK

Background. Several anatomical issues remain unresolved with the current resources of study. Myocardial architecture has been debated concerning the three-dimensional orientation of the cardiomyocytes [1]. Another example relates to the perivascular spaces of the spleen, which has been little studied despite their clinical role in post-traumatic haemorrhage [2]. Through these two examples, we can show the contribution of the hierarchical phase-contrast tomography to the anatomical study.
Methods. Organs were obtained from two bodies donated to the LADAF. After evisceration, organs were post-fixed before being mounted with agar-agar ethanol gel preparation. After degassing, the imaging was performed at the ESRF (BM05 and BM18 beamlines) with scans at 2, 6.5, and 25 µm per voxel [3]. Three-dimensional reconstructions were obtained by manual virtual segmentation using ITK-SNAP Software.
Results. The imaging used enabled us to explore the myocardial architecture by visualizing the fascicles. At first sight, myocardium seemed to be organised as a homogenous continuum, in which every cardiomyocyte sets up in irregular fascicles. These fascicles were aligned in series and coupled to each other through multiple offsprings, the overall meshwork being supported by a matrix of connective tissue. The acute margin of the heart is mainly composed of circumferential fascicles that reach the diaphragmatic part of the heart. Regarding the spleen, we compared its perivascular architecture with that of the kidney, traumatized or not. Unlike the kidney, the spleen appeared to have a space. A double mesh structure was seen with myocytes in the connective tissue surrounding the organ. Medium and small calibre vessels were surrounded by a sheath going deep into the spleen which may explain the non-diffusion of the haemorrhage in the spleen.
Conclusion. This anatomical descriptive report was based on a high-resolution, non destructive approach through synchrotron X-ray imaging of ex-vivo human organs. We prove that this imaging technique allows the assessment of detailed micro-anatomy by addressing previously unresolved issues, i.e. the myocardial organisation with a questioning of the prevailing theories and the peri-vascular spleen organisation with a potential explanation of a misunderstood clinical event.

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Cinematic Visualization: From Organs to Cells and Physiology

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The Cinematic Rendering technology [1] introduces photorealistic 3-dimensional medical imaging to the development of next generation medical research imaging technologies and helps to bridge the gap between macroscopic and microscopic imaging modalities. In doing so, the technology is fostering research that will create a foundation for completely new approaches to teaching [3], patient communication and surgery planning.

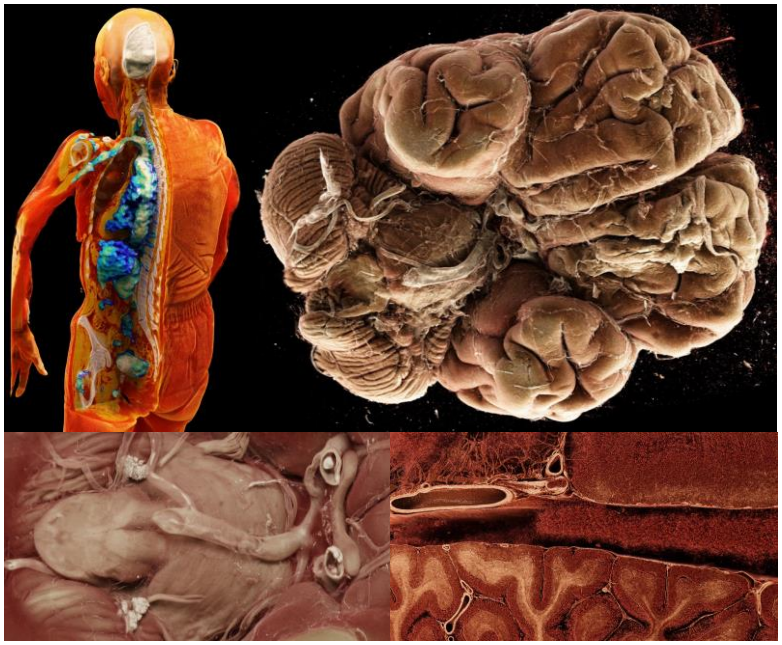

Figure 1: Upper left: FAPI-PET CT visualization allows to differentiate fibrotic from inflammatory processes, Upper right: overview-scan of Hip-CT data from ESRF with zooms into details (lower images).

Handling and visualizing terabyte scale volume data on standard PCs is an enormous challenge. It can be tackled by using out-of-core multi-resolution volume rendering technologies such as CERA-TVR[2]. Combining Cinematic Rendering and out-of-core multi-resolution rendering technologies with novel next generation imaging technologies facilitates the creation of human anatomical atlases, allowing zooms from a full body view to organ systems and organs to the cellular and physiological level. This will leverage the understanding of the human anatomy and physiological processes for researchers, practitioners, and everyone. This talk will introduce these technologies and present results with the latest Hip-CT organ data from the ESRF.

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Industrial Computed Tomography at the BM18 beamline

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The year 2022 marks the inauguration of the world's first industrial tomography beamline for large objects. An offspring of the recent upgrade towards the Extremely Bright Source (EBS), the BM18 beamline was expressly designed for making use of the beam coherence, size and high-energy flux [1,2]. The source being a three-pole wiggler in a vertically low beta-section emits a horizontally broad X-ray beam comprising usable photon energies up to 400keV. Thanks to the exceptionally small source (10µm) and the resulting partial spatial coherence it is convenient to set the source-to-object distance to 180m. At this position the horizontal beam size is 300mm wide, favoring pixel sizes up to 25µm which lead in turn to the development of a novel large area detector design (LAD). For fully realizing propagation-based polychromatic phase contrast, the experimental hutch extends 40m along the beam axis. A motorized slide hosting 9 indirect X-ray detectors with different optics, hence different pixel sampling (from 0.6µm to 42µm) allows for setting the object-to-detector distance continuously between 4m and 36m. The current sCMOS cameras feature 2048^2 pixels resulting in CT slices of 3680^2 voxels if field-of-view extension by half scanning is used. The development of the LAD in particular leads to much larger slices: 27.500^2, hence 1.4Gb/slice in uint16 format (again, field-of-view extension by half scanning is used). For these slices, fast hierarchical back-projection (HBP) was implemented on GPU-workstations thus keeping pace with the camera's high frame rate of 100fps. Following this development, a novel open and "virtually lossless" compressed data format is being developed which will grant the users approx. 12x compression rates as well as local decompression and visualization for accessing and storing these gigantic volume images.

During first explorative experimental campaigns, a large variety of industrial objects were scanned. In many cases, 42µm voxel size were used for obtaining an overview over the entire object, followed by a series of region-of-interest scans of decreasing voxel size, if required down to 0.6µm/voxel (i.e. resolving the fibers in carbon composites or the Nickel-Lithium-Manganese-Oxide particles in Li-ion pouch cells). Results of these explorative campaigns not only revealed that synchrotron CT on BM18 is much surpassing laboratory high-energy CT in terms of contrast and spatial resolution, but BM18 is also outperforming other synchrotron beamlines in these categories thanks to its exceptionally strong propagation-based phase contrast. The sequential acquisition of region-of-interest scans of decreasing voxel sizes has been developed recently during the investigation of pathological lungs (Covid research) and Human-Organ-Atlas (HOA) project. This mode is referred to as Hierarchical Phase-Contrast imaging [1]. Part of the ongoing BM18 project which is funded by the German Federal Ministry of Education and Science (BMBF) under the grant title "05E2019", is dedicated at further developing the interactive choice and visualization of the scanned regions within larger objects.

In conclusion, the BM18 project, which is still ongoing, being the world's first, partially coherent tomography beamline for scanning large industrial (and of course scientific) objects, will achieve several milestone developments. Firstly, a large area detector which may produce 1.4Gb CT slices. Secondly, the semi-automated acquisition of Hierarchical Phase-Contrast scans reaching down to 0.6µm sampling and thirdly, a novel, compressed, virtually lossless data format which guarantees the easy access and handling of the extremely large volume images.

Figure 1: Synchrotron CT scan of a high-grade mechanical wrist watch (18.5µm/voxel).

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Addressing industrial challenges in tomography using BM18 beamline

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Tomography has become a key characterization technique in nearly all industry sectors. In many cases it is now fully integrated in R&D processes and control procedures. At the interface between the synchrotron facilities and industry, Novitom is acting, hand to hand with ESRF, to provide ready-to-use results by operating world-class performance tomography beamlines such as BM05 or ID19.

With the new outstanding capabilities of BM18 beamline, the realm of possibilities has been expanded. Industrial applications will directly benefit from the high quality of tomography images thanks to improved phase contrast and from the possibility to image large samples at high energy and great resolution.

BM18 was used by Novitom over the past year to provide answers to industry collaborators in fields with significant societal impact, particularly in the areas of energy, pharmaceuticals, and medicine. Although many of these projects are subject to confidentiality, some examples can be shared and will be presented. Important technical demands for industrial applications will also be presented such as reproducibility, high throughput capabilities ... Finally, the results obtained from a synchrotron tomography simulation software developed by Novitom (Novi-Sim) to simulate BM18 data collection pipeline and how it can be used both for acquisition parameters optimization and data analysis will also be presented.

Opportunities for Multi-Scale Imaging in Advanced Batteries

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Li-ion batteries have transformed modern life through their ubiquitous application in consumer electronics. Indeed, the revolution in mobile computing would not have been possible without the lithium battery which has consistently delivered improvements in energy and power density commensurate with demands for shorter charge times, and longer battery life for increasingly power-hungry consumer electronics applications. In the future, advanced batteries promise to have an increasing role in our lives, spanning mW to MW applications, with use cases as diverse as medical implants, grid-scale storage, automotive powertrains and short haul flight. In the near to medium-term, it is the Li-ion battery that will meet these requirements, however, with increasingly demanding applications, and diminishing returns in battery performance roadmaps, a range of key challenges persist. This motivates characterization and modelling that is both multi length- and multi-time scale with in-situ/operando capabilities.

Multi-scale imaging is required to describe particle, electrode and cell architectures, and effectively couple information on the material morphology, with electrochemistry and crystallography. X-ray methods are uniquely flexible in their non-destructive and penetrative nature and operate effectively across multiple spatial and temporal domains.

Our previous works have effectively demonstrated the capacity to utilise 3D X-ray tomography to image particles, electrodes and cells; however, it is notable that to date these investigations have been largely siloed at each discrete length scale. A continuum understanding of the constituent materials, cells and packs is required and there is an emerging 'pipeline' for multi-scale X-ray imaging, from the particle (using nano- and micro-CT) to the cell level (using macro-CT), and to correlate the morphology of materials, and architecture of cells to key performance and lifetime metrics.

With the growing industry trend towards larger format cells, there is an opportunity to use novel scanning approaches, including laminography and hierarchical tomography (with extreme region of interest and phase contrast) to assess features buried within large format devices. The latter has been recently demonstrated for imaging full organs from Covid infected patients and has achieved remarkable results in imaging high resolution features buried within macroscopic samples. The same approach for battery science and to visualise electrode level morphological features inside extremely large samples would prove transformative to battery science and engineering, informing new scientific understanding alongside evaluating new cell design and control strategies.

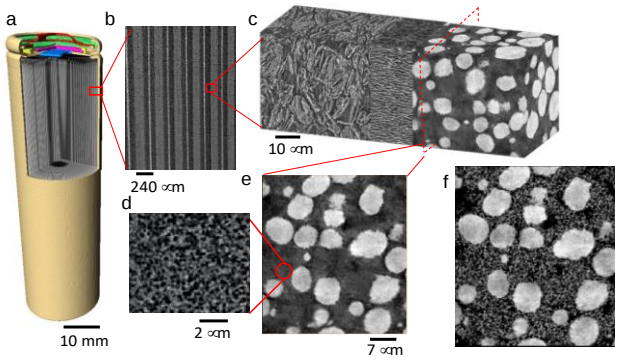

Figure 1: Multi-scale imaging of the cell architecture (a,b) electrode morphology (c,e) and non-active materials (d) which can be effectively registered and super-imposed (f) to combine multi-scale data (*image from X. Lu et al, Nature Comms*).

Performance and Transmitter-Receiver Trade-offs in Giant Frog Acoustic Communication

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Giant anuran species express a morphology with hyper ossification, a large head (which impacts the distance between the inner and outer ear), and a larynx that may be as long as the head as, for example, in Pipa pipa. There is currently no theoretical model that can explain the variety of changes in form and function observed with increasing size. Nevertheless, studies on the physiology of body size [1-3], the evolution of body size [4], size and functional morphology [5], and a general theoretical model [6] show the importance of body size as a biological variable. These animals reach their large size and yet do not have extremely low song frequencies if one follows a linear regression of song frequency against size. On the contrary, they have song frequencies for communication equivalent to species between 5 and 10 times their size. The objective of this study is twofold: 1. how did their vocal system (lung, larynx, oral cavity) evolve? 2. How did their ears develop and how are they adapted to achieve accurate sound source localization, at frequencies equivalent to species 5 to 10 times their size? For comparison, humans can resolve a maximum interaural delay for an average DTI of 800 µs [1]. Acoustic communication is an evolutionarily important trait as it is crucial for courtship behavior and thus for reproduction [7]. We aim to identify and quantify in detail the cartilage, tympanic membrane, bone, and musculature of structures related to sound reception. The definition of the biomechanical properties of the tissues by MDT and the precise morpho-anatomy of the organs have shed light on the understanding of the functioning of sound reception in vertebrates. In frogs (7500 species), a few tympanic middle ear models have been reconstructed in 3D for detailed functional analysis. This study on BM18 allowed us to reconstruct 3D models of giant frogs to explain the various morphologies of the ear from a biomechanical and evolutionary point of view. Specimens were scanned in a 9 to 18 cm field of view with a voxel size of 25 and 42 µm respectively and in phase contrast using techniques developed through the UCL and ESRF beamtimes md1252/90 funded in part by grant number 2020-225394 from the Chan Zuckerberg Initiative DAF, an advised fund of Silicon Valley Community Foundation., and *grant number CZIF2021-006424 from the Chan Zuckerberg Initiative Foundation* [8]. This method on BM18 enabled us to have realistic models which would be used to make physical simulations in order to test hypotheses on the auditory system.

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Using BM18 for multi-scale tomography of large fossils: the pelvic region of the near-tetrapod *Elpistostege*

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Even before the refurbishment, ESRF was unrivalled as a tomographic facility for fossils because of its unique ability to perform propagation phase contrast synchrotron microtomography (PPC-SRμCT) on relatively large specimens [1]. These scans were carried out principally at beamline ID19. However, the construction of the dedicated tomography beamline BM18 has raised these capabilities to an entirely new level, in terms of the maximum size of specimen that can be accommodated, the ability to perform multi-scale scans without remounting the specimen, and the propagation distances that can be achieved. As an illustration of its performance, I present here ongoing work on the pelvic region of the near-tetrapod *Elpistostege*, based on a series of scans made at BM18 in September 2022. *Elpistostege* is a member of the Elpistostegalia, a small group of Devonian fossil fishes that form the immediate sister group to tetrapods (land vertebrates) and are thus potentially informative about the beginning of the fish-tetrapod transition. The single most informative elpistostegalian fossil is a near-complete *Elpistostege* from Miguasha, Quebec, Canada [2], which I am studying in collaboration with Richard Cloutier (Université de Quebec a Rimouski) and Vincent Fernandez (ESRF). We have been scanning a block from the pelvic region, approximately 25 cm long, which contains the pelvic fins and well-preserved contents of the hindgut. The results will thus allow us to investigate both the locomotory morphology and the diet of this important fossil. The scans, done at three different resolutions without remounting the specimen, are of very high quality and allow for easy segmentation of the anatomy. The outer surface of the squamation, which has been hidden in the rock because the specimen is split through the body, can be visualised for the first time and reveals unexpected regionalisation. The pelvic fin skeletons can be 'dissected' out from overlying scales on the basis of microanatomical differences, and the contents of the gut can be itemised. These capabilities greatly increase the information we can extract from *Elpistostege*, and open the door to a new approach to studying large vertebrate fossils.

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How synchrotron scanning is changing the evolutionary origins of mammals

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Mammals are descended from reptilian-looking ancestors, often referred to as the "mammal-like reptiles", which dominated the Earth millions of years before the dinosaurs. For a long time, it has been believed that these "mammal-like reptiles" were primitive: they had unrefined sense organs, a small brain, simple behaviour and were cold-blooded (ectothermy). In the last 10 years, however, synchrotron and lab CT studies of dozens of fossils have demonstrated that their biology and physiology were not so simple.

Recent research on the previously out-of-reach fossil evidence and internal neurological structures of "mammal-like reptiles" supports that modern behaviour such as parental care, burrowing and gregariousness, and the presence of defining mammalian traits such as whiskers, an enlarged brain and warm-bloodedness (endothermy) had already evolved in this lineage well before it gave birth to mammals.

So far, these works were conducted on a selected sample of relatively small fossils, thus limiting the scope of these results. But as they dominated terrestrial ecosystems, "mammal-like reptiles" often reached body sizes that rivaled those of the largest terrestrial mammals, making their CT study challenging. We hope that the new BM18 will provide data about these overlooked large animals (for the first time, non-destructively) and help unravel the exciting complexity of "mammal-like reptiles" biology, physiology and behavioural diversity.

GEOSCIENCES AT BM18: Earthquakes, Volcanoes and CO_2 storage

B. Cordonnier[2,1] on behalf of the Geobridge members and users

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Being natural hazards or societal challenges, geosciences addresses vital yet natural (subsequently complex) phenomena. Where the geological community used opaque autoclaves to reproduce Earth conditions of pressure and temperature, the synchrotron directly observes the studied processes. With the recent EBS of ESRF, new capabilities are unlocked in terms of scanning speed or apparatuses thickness, allowing us to explore larger samples at greater depths. We develop on beamline BM18 several samples environments capable of reproducing either pressure, temperature or fluid flow encountered in the Earth's crust's first kilometres. These environments aim to provide the first quantitative laboratory for geological problematics. Today's discussion illustrates direct applications of this new fleet of geo-devices with an example for earthquake mitigation, volcanic hazards and CO2 storage.

In volcanic structures, flow or blow are the two primary outcomes. When the magma viscosity can no longer diffuse the excess pressure fast enough, the magma shatters, and its previously flowing behaviour transforms into catastrophic explosions. A high-temperature furnace is under test to understand the complex three-phase rheology interaction of flowing magmas. Ultimately coupling decompression and temperature will allow us to investigate the whole spectrum of volcanic dynamics. Still, the gas phase interaction and network development already provide critical insight into magmatic ascent and dynamics.

The second setup presented investigates rock permeability and fluid flow under reservoir conditions. Today, relieving the carbon dioxide which saturates our atmosphere and aggravates the ongoing climate change is a priority. Therefore, a few large-scale initiatives attempt to capture atmospheric CO2 to inject it into the ground and turn it into rocks. Again, the ESRF may help understand this chemical reaction and improve its efficiency. Preliminary tests are performed on potential rock candidates to understand their structure, properties and how the saturated CO2 fluids interact with them.

Finally, regarding the earthquake mechanisms, the principal investigator, François Renard, obtained a competitive European Research Council (ERC) Advanced Grant "Break through rocks" for 2022-2026. It allowed the development of the tri-axial press '*Zeus*'. A transparent device capable of recording acoustic emissions produced during the rupture of rocks while imaging rock integrity. Deformation in the Earth's crust localises onto faults that may rupture rapidly, producing earthquakes, or undergo slow aseismic slip that may or may not accelerate into a coseismic slip. However, the detailed mechanisms that control the transition between the seismic and aseismic regimes and the onset of earthquakes remain unknown. Zeus, acquiring simultaneously dynamic synchrotron X-ray microtomography and acoustic emission, will generate novel experimental techniques that allow us to separate the aseismic and seismic deformation components. The data will reveal how slow and fast deformation processes develop and interact with each other in dry and wet crustal rocks under stress, fluid pressure, and temperature conditions at depths up to 10 km and characterise fracture network development during earthquake nucleation and rupture propagation.

Correlative imaging for additive manufacturing of metallic materials

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Additive manufacturing of metallic alloys is nowadays heavily investigated by the academic and the industrial community. Nevertheless many process parameters can affect the soundness of the built parts. The resulting mechanical performances are driven by features at entangled characteristic scales (microstructure, melt pool, layer, struts, part…). Multi-scales 3D characterizations, in situ or post-mortem, are valuable to undisclose defects genesis and to drive the optimisation of the process.

At the scale of the melt pool, the microstructure can be highly heterogeneous with a complex 3D ordering of equiaxed and columnar grains. A fine 3D characterization resolved enough to describe the grains shape and size but covering a field of view large enough to capture a complete melt pool profile enable to enravel the complexity of the microstructure. ID16B is particularly suitable for this purpose with multi-scale in situ nano-tomography experiments [1].

At the scale of the part, the high X-ray attenuation induced by high temperature applications alloys (such as Ni based superalloys) and the size of the built parts (several tens of centimeters) makes X-ray imaging impossible on a standard tomography beamline. The potential of BM18 for such application is revealed with few preliminary acquisitions.

At intermediate scale (the scale of the strut, the scale of a bulk portion, the scale of a few adjacent and stacked melt tracks), a comprehensive view on defect formation and healing is seek for. Complementary to radioscopy observations on 2D builds, an additive manufacturing replicator compatible with X-ray micro-tomography [2,3] enables to image in operando the building process of small 3D parts (cubes, small lattices…) as exemplified with acquisitions conducted on BM05.

The long term project MA4928 : Correlative Imaging for Additive Manufacturing of Metallic Materials (CIAM3), encompasses all these scales. This LTP, shared between ID16B and BM18, aims at offering a multiscale characterisation suitable for the investigation of metallic additive manufacturing. It consists in a dual strategy :
(i) an experimental strategy combining and linking in situ tests at several scales: In situ tests cover a wide range of the sub process involved in the investigated AM technologies and scientific cases : constrained sintering, 3D printing, mechanical properties characterization;
(ii) a numerical strategy based on trained Artificial Intelligence (AI) algorithms: they will allow performing automatic data reduction, acquisition with lower number of projections, automatic noise and artefact removal. Spatial resolution of low resolution reconstruction would be increased thanks to training on high resolution acquisitions. The ability to recover high resolution information on low resolution acquisitions would allow to significantly increase the ROI/feature size ratio.

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User-Dedicated Microsymposium UDM 3

8 February 2023

Operando science of functional energy conversion, storage materials and devices

UOC Organiser	Karsten Küpper Alberto Martinelli
ESRF Organisers	Jakub Drnec Marta Mirolo
Non-ESRF Organiser	Raphael Chattot Quentin Jacquet

- Programme
- Abstracts of Keynote & User Talks

UDM3. Operando science of functional energy conversion, storage materials and devices

Wednesday, 8 February 2023 - Microsymposium UDM3 ESRF Auditorium	
8:50 – 9:00	**Welcome from the organisers**

Morning session

	Session I - Chair: R. Chattot	
9:00 – 9:30	**Keynote talk 1** The Use of Operando Scattering to investigate the Degradation of Pt/C Fuel Cell Catalyst	**Matthias Arenz** *University of Bern*
9:30 – 9:50	**User talk** *Operando* XAS Analysis of Co-Fe co-Catalysts in a Flow Photoelectrochemical Cell	**Raffaello Mazzaro** *University of Bologna*
9:50 – 10:10	**User talk** *Operando* High-energy Surface X-ray Scattering Studies of Oxide Growth and Surface Restructuring of Pt Single Crystal Electrodes	**Olaf Magnussen** *Kiel University*
10:10 – 10:30	**User talk** Breathing Pt nanoparticle imaged using in situ BCDI	**Frederic Maillard** *INP Grenoble*
10:30 – 11:00	*Coffee break*	
	Session II - Chair: A. Martinelli	
11:00 – 11:30	**Keynote talk 2** Advancing Materials for CO_2 Capture and Conversion: Insights from Structural Characterization under Operating Conditions	**Paula Abdala** *ETH Zürich*
11:30 – 11:50	**User talk** Tandem CO_2 Valorization and Ethane Dehydrogenation: Nature of Highly Selective Iron Oxide Active Sites and the Low Z Elements Point of View	**Alessandro Longo** *ESRF Grenoble*
11:50 – 12:10	**User talk** Structural Defects Related to Carbon Contamination during the Solidification of Silicon for Photovoltaic Applications: *ex-situ* and *in situ* X-ray Imaging Characterization	**Nathalie Mangelinck-Noël** *Aix Marseille University*
12:10 – 12:30	**User talk** Multimodal Characterization of $LiNiO_2$/Graphite Batteries: Going towards Understanding Lithiation Heterogeneities at High Cycling Rates	**Quentin Jacquet** *CEA Grenoble*
12:30 – 13:50	*Lunch Break*	

UDM3. Operando science of functional energy conversion, storage materials and devices

Afternoon session

	Session III - Chair: M. Mirolo	
13:50 – 14:20	**Keynote talk 3** Understanding Battery Electrochemistry by Bathing Model Systems in (Coherent) X-Rays	**Hans-Georg Steinrück** *Paderborn University*
14:20 – 14:40	**User talk** The Importance of Experimental Conditions in *Operando* Batteries Studies: the Case of $LiNiO_2$ Cathode Material	**Antonella Iadecola** *RS2E Amiens*
14:40 – 15:00	**User talk** Beam Damage in *Operando* Synchrotron Studies: Conditions and Consequences	**Thibaut Jousseaume** *CEA Grenoble*
15:00 – 15:20	**User talk** Energy Dispersive EXAFS Studies on Rechargeable Batteries at the ODE Beamline Soleil Synchrotron	**Qingyu Kong** *Synchrotron Soleil Gif-sur-Yvette*
15:20 – 15:40	**User talk** *In Situ* Measurements on Battery Cells with X-ray Nano-Tomography	**Olga Stamati** *ESRF Grenoble*
15:40 – 16:00	**User talk** XRD-CT Investigation of LPSCl Degradation Occurring during Cycling	**Oskar Thompson** *LEPMI Grenoble*
16:05	**Closing remarks and farewell**	

The Use of Operando Scattering to investigate the Degradation of Pt/C Fuel Cell Catalyst

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Proton exchange membrane fuel cell (PEMFC) powered vehicles are a promising alternative to combustion engines in the heavy-duty sector. For such applications extended lifetimes of the electrocatalysts are required. To study the degradation behavior of PEMFC catalysts typically accelerated stress tests (ASTs), simulating driving conditions are applied and the response of the catalyst is investigated. Local methods such identical location transmission electron microscopy (IL-TEM) provide information of the degradation on a local scale[1], however, often several different degradation mechanisms occur. To investigate the overall behavior of the catalysts, therefore integrative methods such as small- and wide-angle scattering (SAXS and WAXS) are preferred. Furthermore, these techniques can be applied operando, i.e., during the AST. In the presentation, I will present our work on operando SAXS/WAXS degradation studies[2,3] and highlight advantages, but also the challenges in coupling these techniques to electrocatalytic measurements.

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Operando XAS Analysis of Co-Fe co-Catalysts in a Flow Photoelectrochemical Cell

R. Mazzaro[1], A. Piccioni[1], L. Pasquini[1], S. Caramori[2], A. Puri[3], F. D'Acapito[3], F. Boscherini[1]

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Water oxidation photoanodes based on earth-abundant metal oxide semiconductors are actively studied due to their limited environmental impact, the stability to photo-corrosion and the tunable optoelectronic properties [1]. $BiVO_4/WO_3$ heterostructures are now emerging as one of the most promising photoanode, but the activity is limited by surface recombination and sluggish charge transfer kinetics. Cobalt-iron mixed compounds, such as CoFeOx and CoFe hexacyanoferrate, a Prussian blue analogue, proved to effectively enhance photocurrent when coupled to $BiVO_4/WO_3$ photoanodes as water oxidation co-catalyst. The origin of the increased efficiency is still debated, but recent studies suggest an enhancement of charge separation efficiency within the semiconductor [2], rather than improved water oxidation kinetics, underlying possible charge accumulation on the co-catalyst leading to transient modification of the local structure. To achieve a thorough understanding of the semiconductor and catalyst structure in the actual PEC operating conditions, a custom cell was specifically designed to allow for XAFS analysis with a thin electrolyte layer (<100 μm) in continuous flow, preventing bubbles formation and enhancing mass transfer (Fig 1a). The effect of sunlight-equivalent illumination and bias, fully integrated with the beamline apparatus (LISA BM-08) and remotely controlled, was investigated by either performing fixed potential spectroscopy (Fig 1b), or fixed X-ray energy absorption voltammograms [3] (FEXRAV, Fig 1c). A tailored algorithm for point-by-point spectral acquisition triggering light stimulus was also implemented to achieve time-gated acquisition and compensate drifts in the acquisition setup over long-term measurements. The resulting characterization displayed a drastic modification of the structure between operando and ex-situ analysis, as well as a dependence on bias and illumination, further investigated by EXAFS analysis.

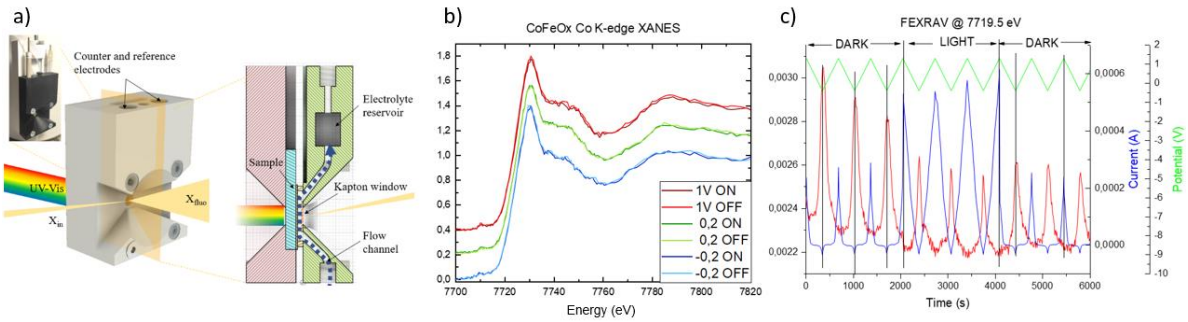

Figure 1: a) 3D-printed custom PEC cell scheme; b) fixed potential XAS spectra in dark/light conditions at Co K-edge; c) FEXRAV @7719.5eV in dark/light conditions.

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Operando High-energy Surface X-ray Scattering Studies of Oxide Growth and Surface Restructuring of Pt Single Crystal Electrodes

T. Fuchs[1], J. Drnec[2], J.O. Fehrs[1], C. Yuan[3], D.A. Harrington[3] and O.M. Magnussen[1]

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The need for sustainable energy, reduction of pollutants, and the environmental benign processing of chemicals has spurred worldwide scientific activities in electrochemical energy science and electrocatalysis. These processes occur at the interfaces of solid catalyst materials in contact with complex liquid environments and under conditions involving high reaction rates, pronounced mass transport, and vigorous gas evolution. We here describe high-energy X-ray scattering methods for *in situ* and *in operando* studies that provide insight into the complex interfaces between electrocatalysts and liquid electrolytes under reaction conditions. The application of these methods is demonstrated for the case of electrochemical Pt surface oxidation, which is of key importance for the development of durable oxygen reduction reaction catalysts as used in low temperature fuel cells. In this process, the formation of an ultra-thin oxide film on the electrode surface is causing atomic-scale restructuring and Pt dissolution, which promotes the degradation of the Pt catalyst.

By High Energy Surface X-ray Diffraction measurements at ID31 of the ESRF and P21.2 of PETRA III, we analysed the atomic-scale surface structure of Pt(111) and Pt(100) during oxide formation and reduction in 0.1 M $HClO_4$ and 0.1 M H_2SO_4 [1]. These surfaces exhibit distinct differences in stability versus restructuring after potential cycles to the same upper potential limit (Fig. 1a, b). To elucidate this difference we performed a detailed analysis of the crystal truncation rods at different potentials from the onset of oxide formation up to the onset of oxygen evolution to determine the location and potential-dependent coverage of the Pt atoms in the oxide. This revealed distinct differences in oxide growth mechanism on Pt(111) and Pt(100) which explain the differences in structural stability. Repeated surface oxidation and reduction leads to growth of Pt nanoislands (Fig. 1d), which were studied using Grazing Incidence Small Angle X-ray Scattering. The island growth was analysed as a function of oxidation potential, number of potential cycles and surface structure, indicating a qualitatively similar changes of the nanoscale morphology but distinct differences in the quantitative roughness evolution.

This work was funded by the Deutsche Forschungsgemeinschaft via grant 418603497 and the BMBF via project 05K19FK3.

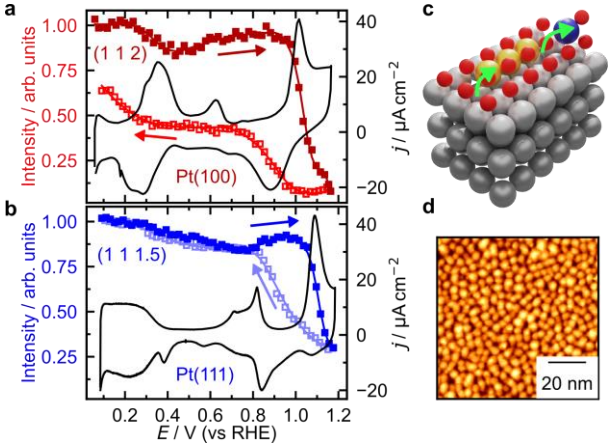

Figure 1: X-ray intensity and electrochemical current density during potential cycles on Pt(111) **a,** and Pt(100) **b**, with 20 mV/s. Oxide formation is indicated by the decrease of the intensity at about 1 V. **c,** Illustration of the surface oxide on Pt(100) [4]. **d**, STM image of the Pt nanoislands grown on Pt(100) after 50 ox./red. cycles to 1.62 V.

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Breathing Pt Nanoparticle Imaged using *in situ* BCDI

C. Atlan[1,2,3], C. Chatelier[1,2], I. Martens[2], M. Dupraz[1,2], S. Leake[2], M.-I. Richard[1,2], A. Viola[3], F. Maillard[3]

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The relationship between surface strain and the rate of a (electro)catalytic reaction was unveiled by Hammer and Nørskov using density functional theory (DFT) calculations [1,2]. They proposed that the rate of the sluggish oxygen reduction reaction (ORR) would be enhanced on Pt-based catalysts binding *OH species *ca*. 0.10 - 0.15 eV more weakly than Pt(111), [1, 2] later experimentally verified using a $Pt_3Ni(111)$-skin surface [3]. Nevertheless, these predictions did not translate to Pt-based nanocatalysts, in part because these present multiple catalytic sites with a wide range of binding energies. Hence, an *in situ* picture of how strain is distributed on Pt-based surfaces is still lacking.

In this contribution, we took benefit of recent advances in Bragg Coherent Diffraction Imaging (BCDI) [4, 5] and of the fourth generation Extremely Brilliant Source of the European Synchrotron (ESRF-EBS, Grenoble, France) to map strain over Pt nanoparticles in electrochemical environment. Our results reveal that strain is heterogeneously distributed between highly- and weakly-coordinated surface atoms, and propagates from the surface to the bulk of the Pt nanoparticle as (bi)sulphate anions adsorbed on the surface.

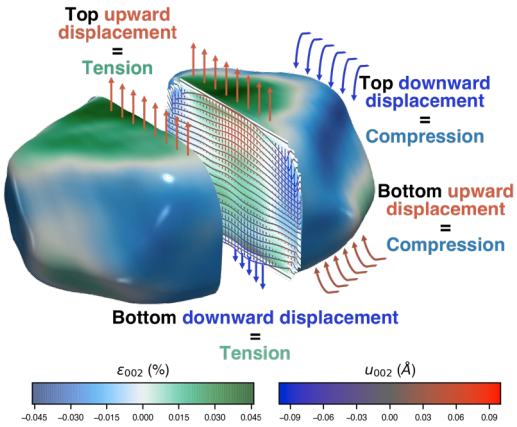

Figure 1: Representation of the strain distribution observed on a Pt nanoparticle under potential control. T = 24°C, 0.05 M H_2SO_4

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Advancing Materials for CO_2 Capture and Conversion: Insights from Structural Characterization under Operating Conditions

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Global concerns about the rising level of greenhouse gas emissions and the related climate change demands the deployments of efficient processes that allow the selective removal of CO_2 from large point sources or directly from the atmosphere. To combat the ever-increasing emissions of CO_2 into the atmosphere, the development of materials (CO_2 sorbents and catalysts) to efficiently capture CO_2 and convert it into value-added chemicals or fuels is key. [1-5] The rational advancement of CO_2-sorbents and catalysts requires an in-depth knowledge of processes taking place at the atomic scale under reaction conditions. This talk will cover a series of studies employing in situ and/or operando synchrotron X-ray based methods to shed light onto the mechanisms at play at the atomic level of CO_2-sorbents and catalysts for CO_2 valorisation. [1-2] Specifically, we will discuss some recent advances in the development of CO_2 sorbents that are based on MgO. [1-2] Moreover, we will focus on structure-performance relationships in catalyst for CO_2 valorisation via the dry reforming of methane or the CO_2 hydrogenation reaction. [3-4]

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Tandem CO_2 Valorization and Ethane Dehydrogenation: Nature of Highly Selective Iron Oxide Active Sites and the Low Z Elements Point of View

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Ethylene is a widely-used petrochemically derived monomer, with a production of 150 million tons in 2018, which is estimated to attain 185 million tons in 2023, due to the population growth and the rising living standards. Nowadays, the non-catalytic steam cracking of naphtha is considered as the dominant process for ethylene production, mainly due to the "economy of scale" [1]. However, the abundance of the stranded gas reserves, combined with the so-called "shale gas revolution", have revitalized the research interest for the employment of underutilized ethane towards the ***"on-purpose" ethylene production routes***. The use of oxygen as an oxidant for the ethane dehydrogenation (EDH) process has been extensively investigated in literature, due to the exothermicity of the reaction [2]. However, the use of oxygen results in low ethylene yields due to the undesired total oxidation reactions towards COx. CO2 can act as a soft oxidant for EDH, shifting the thermodynamic equilibrium through H2 consumption via the reverse water gas shift (RWGS) reaction. Utilization of CO2 from large stationary points is in alignment with EU green deal and can significantly contribute to CO2 emissions curbing towards 2050. Iron- and/or nickel- oxide based catalysts, supported on different oxides, have been previously investigated for the tandem reactions of EDH and CO2 reduction elucidating the role of an interface between iron oxide and nickel oxide [3-6]. The latter interface was found to contribute to the enhanced performance. To unravel the nature of active sites that are responsible for the initial catalytic performance, advanced characterization techniques, such as XAS and XRS, along with structural modelling were applied. In our study we report important structural modification of the catalyst and the support. Its modification induced by incorporation of Fe into the support lattice, leading to a structural change of Mg geometry, as it was demonstrated by modelling of XRS data, at Mg L2,3.

Under CO2 - EDH at 650°C, the nature of active sites was also dynamically changing leading to a C2H4 selectivity increase, at approximately constant C2H6 conversion: from ~ 74% at 1h TOS to ~ 90% at 6h TOS. [7]

This results in unprecedented efficiency and selectivity and opens up novel substitution strategies, based on modulating chemistry and the anion-transition metal interactions, as avenues to design new improved catalysts.

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Structural Defects Related to Carbon Contamination during the Solidification of Silicon for Photovoltaic Applications: *ex-situ* and *in situ* X-ray Imaging Characterization

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In order to improve the PV efficiency of crystalline silicon (Si) solar cells, research focuses on several key targets, which include the understanding of structural defect generation during the solidification. Indeed, the industrial processes all face challenges to decrease the defect density, e.g. dislocations and sub-grains, and to understand their interaction with the impurities during solidification to ultimately improve solar cell electrical properties.

Several samples all contaminated with carbon (C) are studied in this work: samples directly extracted from industrial ingots and model samples. The effect of C on the grain nucleation, grain boundary types, distortions and structural defects at several scales is analysed in details *ex situ* and *in situ* during the Si solidification. *In situ* X-ray imaging is performed in a unique device named GaTSBI (Growth at high Temperature observed by X-ray Synchrotron Beam Imaging) operated at ID19/ESRF. Two imaging techniques are combined during solidification: radiography and Bragg diffraction imaging (topography) [1]. They reveal the morphology and the kinetics of the solid/liquid interface, the defect formation and crystalline structure distortion dynamics. Rocking Curve Imaging (RCI) [2] is performed at BM05/ESRF *ex situ* after solidification to characterize quantitatively the crystalline structure distortions and misorientations.

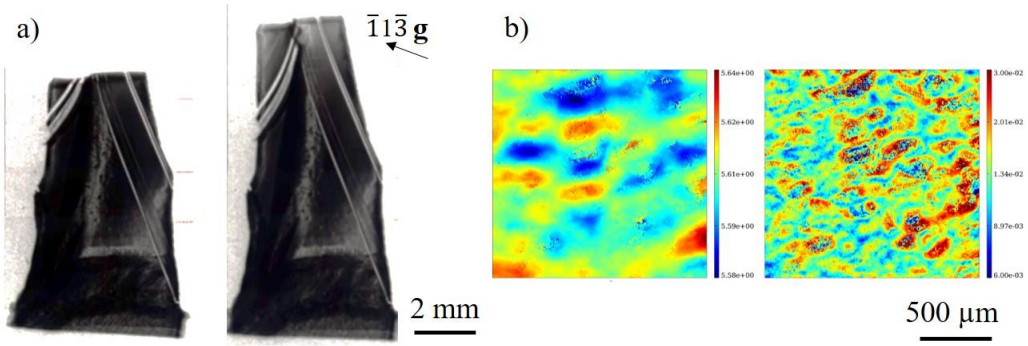

Figure 1: Sample contaminated with C (10^{17} at/cm^3): a) *In situ* topographs at two instants during solidification. b) Transmission RCI maps. Left: Peak-Position, in degree. Right: Full Width Half Maximum, in degree.

Crystal structure distortion is evidenced at several scales both *in situ* and after cooling down in the presence of C. Figure 1 shows topographs during solidification (Fig. 1.a) and RCI peak position and full width half maximum maps (Fig. 1.b) for a sample contaminated with C (10^{17} at/cm^3). We propose a mechanism related to the presence of C during Si solidification to explain the formation of these distortions and of sub-grains in the crystalline structure.

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Multimodal Characterization of $LiNiO_2$/Graphite Batteries: Going towards Understanding Lithiation Heterogeneities at High Cycling Rates

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Mastering fast charging of high energy density Li-ion batteries is key to accelerate market penetration of electric vehicles (EV). This will be achieved by better understanding lithium concentration heterogeneities developing under high current densities, due to limited Li charge transfer rate or diffusion in the porous electrodes[1]. Disentangling these effects is possible through characterisation informed modelling currently under construction in BIGMAP, a European project part of Battery2003+ initiative. Characterisation wise, two different physical properties need to the measured on standardized samples: (1) electrode 3D morphology at nano/micro scale ideally during cycling, (2) spatially resolved lithiation heterogeneities at different charging rates The latest point being particularly tricky because requiring *operando* characterisation capable of quantifying lithium concentration at the micron scale in solid (electrodes) and liquid (electrolytes) phases within minutes[2].

In this talk, we will show Li heterogeneity quantification using correlated *operando* high-resolution neutron imaging (NI) and depth resolved micro-X-ray diffraction (µ-XRD) on a $LiNiO_2$/graphite full cell using the same *operando* cell (see Figure). NI experiments were performed at the NeXT instrument at ILL, and synchrotron data were acquired on the ID31 beamline at ESRF in the frame of the Battery Pilot Hub project. Our correlative data acquisition and analysis method can be extended to other techniques in the frame of new experimental workflows developed in BIGMAP.

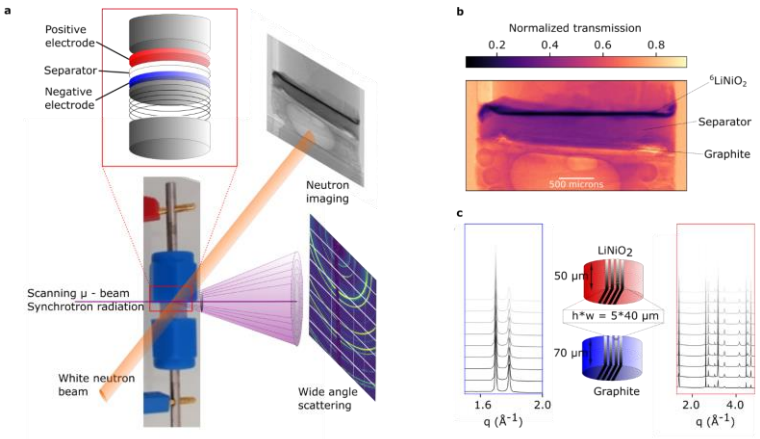

Figure ***a*** Schematic of the correlated NI and µ-XRD experiment, ***b*** typical neutron radiography, ***c*** µ-XRD scanning and typical XRD patterns

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Understanding Battery Electrochemistry by Bathing Model Systems in (Coherent) X-Rays

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The chemistry and physics underlying the functionality of energy materials is complex, but needs to be understood towards knowledge-inspired improvements and novel concepts. Our approach to tackle this challenge is the combination of advanced electrochemical and *operando* X-ray methods with model systems. The objective is a foundational and scalable understanding of the functional role of each atom. Following a brief overview of our approach and recent progress, two examples will be discussed in detail.

The first topic concerns charge and mass transport in electrolytes. We employed a novel approach to understand the ion transport mechanism and evaluate transport coefficients in a baseline polymeric electrolyte. Coherent X-rays were used to measure electrolyte velocity and concentration profiles upon cell polarization. The results were combined with macroscopic concentrated solution theory and microscopic molecular dynamics simulations to quantify and rationalize transference numbers, and to provide length-scale bridging insight into ion transport.

The second topic covers the surface-electrochemistry in Li-ion batteries, with a specific focus on the origin of LiF in the solid electrolyte interphase. Towards this end, we sought out a multimodal experimental and theoretical approach using inert single crystalline model electrodes. We combined operando surface X-ray scattering with voltammetric scans of various electrolyte formulations, and quantum chemical calculations. Our results reveal that LiF nucleates via the electrocatalytic transformation of HF followed by significant PF6- anion reduction.

The final part of the talk will be devoted to future opportunities to utilize coherent and high-brilliance X-rays to study dynamic processes in energy storage materials, in particular using the advantages of the newly commissioned ESRF–EBS.

The Importance of Experimental Conditions in Operando Batteries Studies: the Case of $LiNiO_2$ Cathode Material

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The understanding of the electrochemical reactions occurring in working conditions in rechargeable batteries is essential to design sustainable, safe and low-cost devices with high energy density. Despite the flourish of operando studies, mainly using X-ray synchrotron- and neutron-based techniques, the lack of standardization of the experimental conditions hinders the direct comparison of the results obtained from different experiments on the same material [1]. The definition and application of a standard experimental workflow is one of the pillars of the Battery Interface Genome - Materials Acceleration Platform (BIG-MAP) project [2], that propose to combine large-scale and high-throughput characterization with high performance computing and artificial intelligence approach. In this framework, we present an operando XAS study on the targeted $LiNiO_2$ layered oxide as positive electrode for Li-ion batteries. An in situ electrochemical cell has been specifically developed for combining XAS and Raman spectroscopy, and the preliminary results obtained at the ROCK beamline will be presented.

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Beam Damage in *Operando* Synchrotron Studies: Conditions and Consequences

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Most energy storage devices are taking advantage of the electrochemical reactions time scale ranging from the microsecond to the minute to convert electrical energy into chemical energy [1]. Such a large time scale needs *operando* studies to shed light on the broad nature of mechanisms taking place in these systems. The highly bright synchrotron radiation allows fast acquisitions needed for time resolved studies, and so it is commonly used to probe different kinetics in the battery community.

However, such an intense X-ray beam raise the question of unwanted interactions with the sample caused by photo-absorption or Compton scattering. The side reactions can degrade the battery components and alter the energy storage mechanism under scrutiny. This so-called beam damage has already been reported to be the cause of structure modification in protein crystallography experiments, requiring a close monitoring of the radiation dose [2]. Yet, a few studies report beam damage effects on the battery materials operation [3, 4, 5], and to our knowledge, none are quantitatively evaluating the X-ray dose absorbed with indications to consider when performing an *operando* study.

Here we report a quantitative study on beam damage in Li-ion batteries. We investigate the different possible consequences of beam damage and its onset conditions. To do so, we closely monitor the X-ray dose absorbed by the materials and the electrolyte, and vary it to assess its effect by using a common synchrotron technique: *operando* X-ray diffraction. First, we evidence artificial phase transition triggered by a large X-ray exposure. Comparing it to a sample that has undergone limited irradiation, we reveal that the charge mechanism being probed is skewed by the biased transition. Then, we manage to follow finer structure and microstructure modifications of material evolution provided by an over-exposure to the beam. Based on the precise dose monitoring, we defined several X-ray dose threshold related to a type of material degradation. Finally, we consider the dose rate, which also affects the extent of damages the material is subjected to. Thereby, we unveil that beam damage is driven by the mingled action of dose and dose rate.

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Energy Dispersive EXAFS Studies on Rechargeable Batteries at the ODE Beamline Soleil Synchrotron

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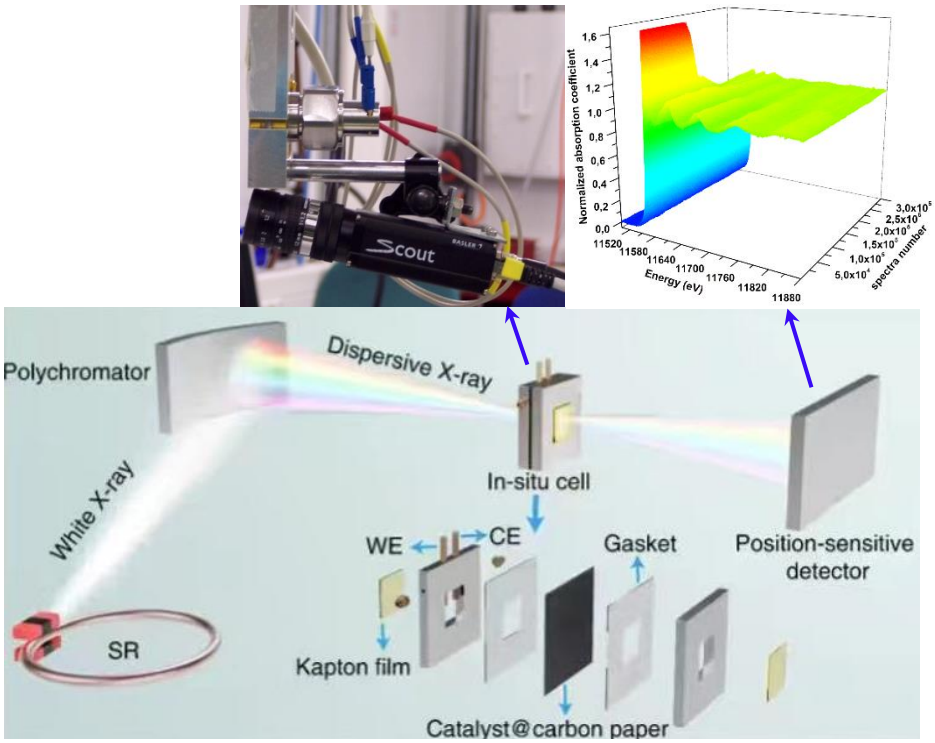
The energy dispersive EXAFS (EDE) setup has been proved to be an applicable technique to study rechargeable batteries as shown in the schematic figure. Different ions (lithium, sodium, magnesium and zink) batteries have been measured with EDE, either in time-resolved operando or static XAS mode at ODE beamline Soleil synchrotron [1-5]. The disadvantage and advantage of EDE on battery studies will be discussed.



Schematic experimental setup for in-situ EDE on rechargeable batteries

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In Situ Measurements on Battery Cells with X-ray Nano-Tomography

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The development of more efficient future energy storage systems requires a deeper understanding of the impact of the internal microstructure on the material properties and the electrochemical performance of the battery cell. This implies the use of non-destructive advanced characterization techniques which can provide high resolution (particle sub-micrometer scale) and sufficient contrast. In this talk, a workflow to 3D nano-image the electrochemical cells in situ will be presented. A nano-tomography in situ setup has been specifically designed to take advantage of the high flux and high coherent X-ray nano-beam provided at the ID16B beamline of ESRF-EBS, combined with the fast acquisition available with the new generation of cMOS detectors.

In particular, the talk will discuss the 3D morphological characterization of the internal structure of electrodes: solid-pore phase identification, interface characteristics, tortuosity, particle/pore size distribution, particle/pore orientation, etc. In addition to the characterization of the initial morphology, the 4D (3D + time) quantification of the progressive evolution of the morphology with cycling will be explored through the measurement of kinematic fields (displacements and strains) in the bulk electrode. These time-resolved measurements offer the possibility to study the performance degradation mechanisms of the cell during operation. The obtained results prove the applicability of the designed workflow at ID16B for in situ studies of electrochemical cells and suggest that future similar applications can be very valuable for the improvement of the performance of existing systems and can potentially lead to a better design of new battery materials.

XRD-CT Investigation of LPSCl Degradation Occurring during Cycling

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Sulphur based solid electrolytes (SE) are a promising candidate for realising solid state battery systems as they exhibit both reasonable ionic conductivity[1] and ease of processability. These solid electrolytes are unfortunately not fully stable upon cycling, due to their narrow electrochemical stability, causing electrochemical degradation during cycling which reduces their long-term electrochemical performance. Despite current investigation, the link between structural and morphological changes and electrochemical performance is yet poorly understood especially during lithiation and delithiation[2].

X-ray diffraction computed tomography (XRD-CT) carried out at ID15a at the European Synchrotron Radiation Facility allows distribution of specific chemical species within a material to be resolved spatially. The focus of this work, in which ex-situ half cells of L_6PS_5Cl have been examined using XRD-CT after 50 charge-discharge cycles against lithium-indium, in both reduction and oxidation and compared to a pristine sample, is to observe the localisation of chemical changes as a function of the depth of the reaction in the cells and gain insight into how they affect the morphology.

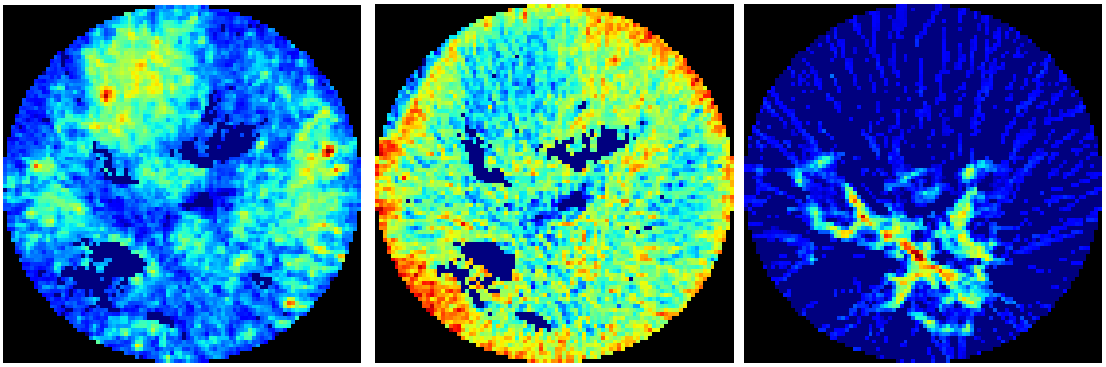

Figure 1: phase distribution maps of (left to right) LPSCl, LiCl and InLi close to the LiIn electrode of a half cell cycled 50 times between 2V and 0.05 V.

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93	Victor Vanpeene, Julie Villanova, Willy Porcher, Isaac Martens, Jérémie Auvergniot, Marion Chandesris, Sandrine Lyonnard	High-resolution X-ray nano-tomography for in-depth battery material analysis

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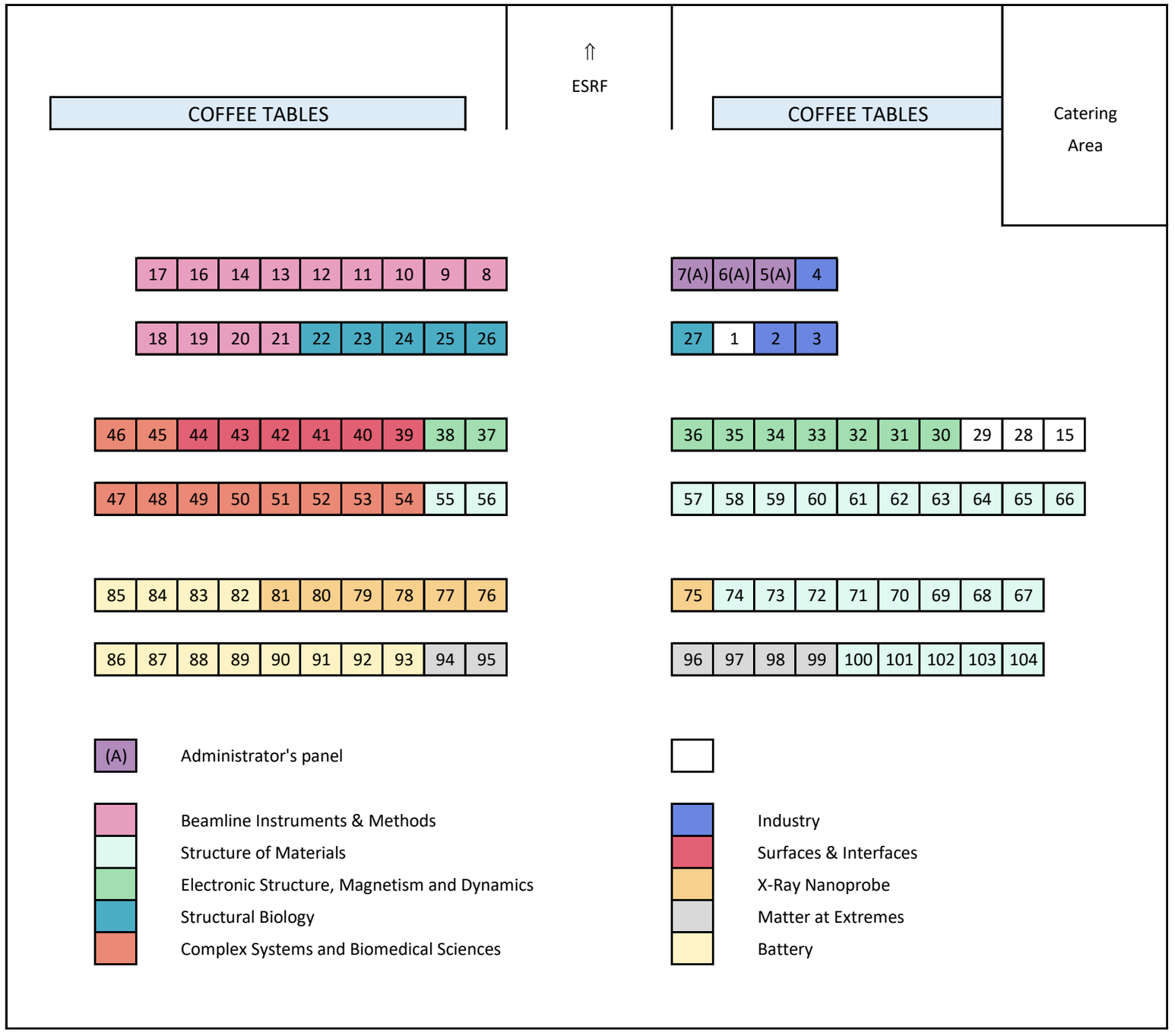
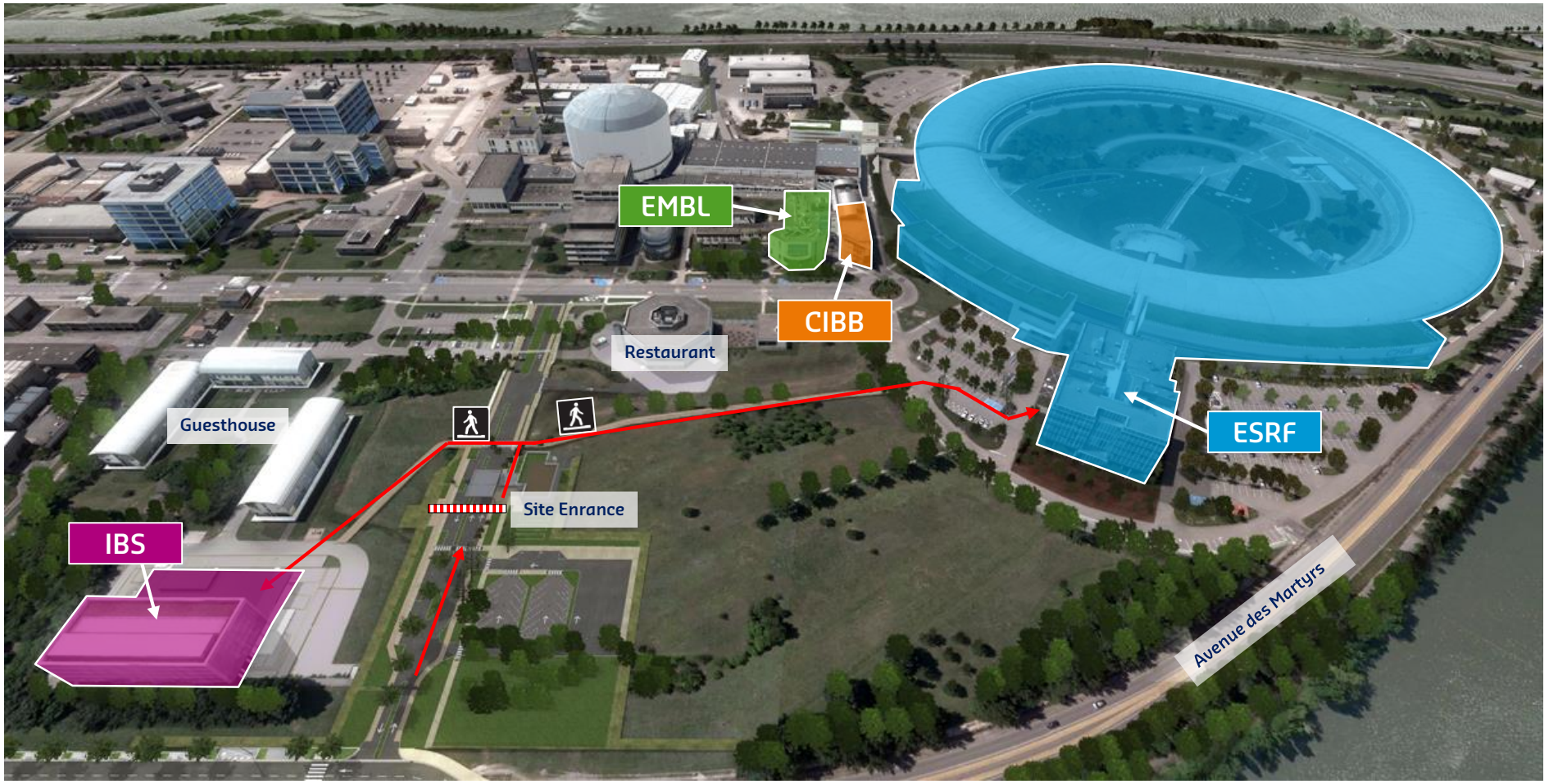
UPDATED Room Plan for Posters



Maps of Venues

- EPN Campus
- Tutorial Venues
- Plenary Session and UDM Venues

EUROPEAN PHOTON & NEUTRON SCIENCE CAMPUS - GRENOBLE



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Shuttle from/to Geneva Airport
Tramway

Highway

To Chambery / Geneva

CIBB	Carl-Ivar Bränden Building – Partnership for Structural Biology
EMBL	European Molecular Biology Laboratory Grenoble
ESRF	European Synchrotron Radiation Facility
IBS	Institut de Biologie Structurale

USER MEETING 2023 – Tutorial Venues

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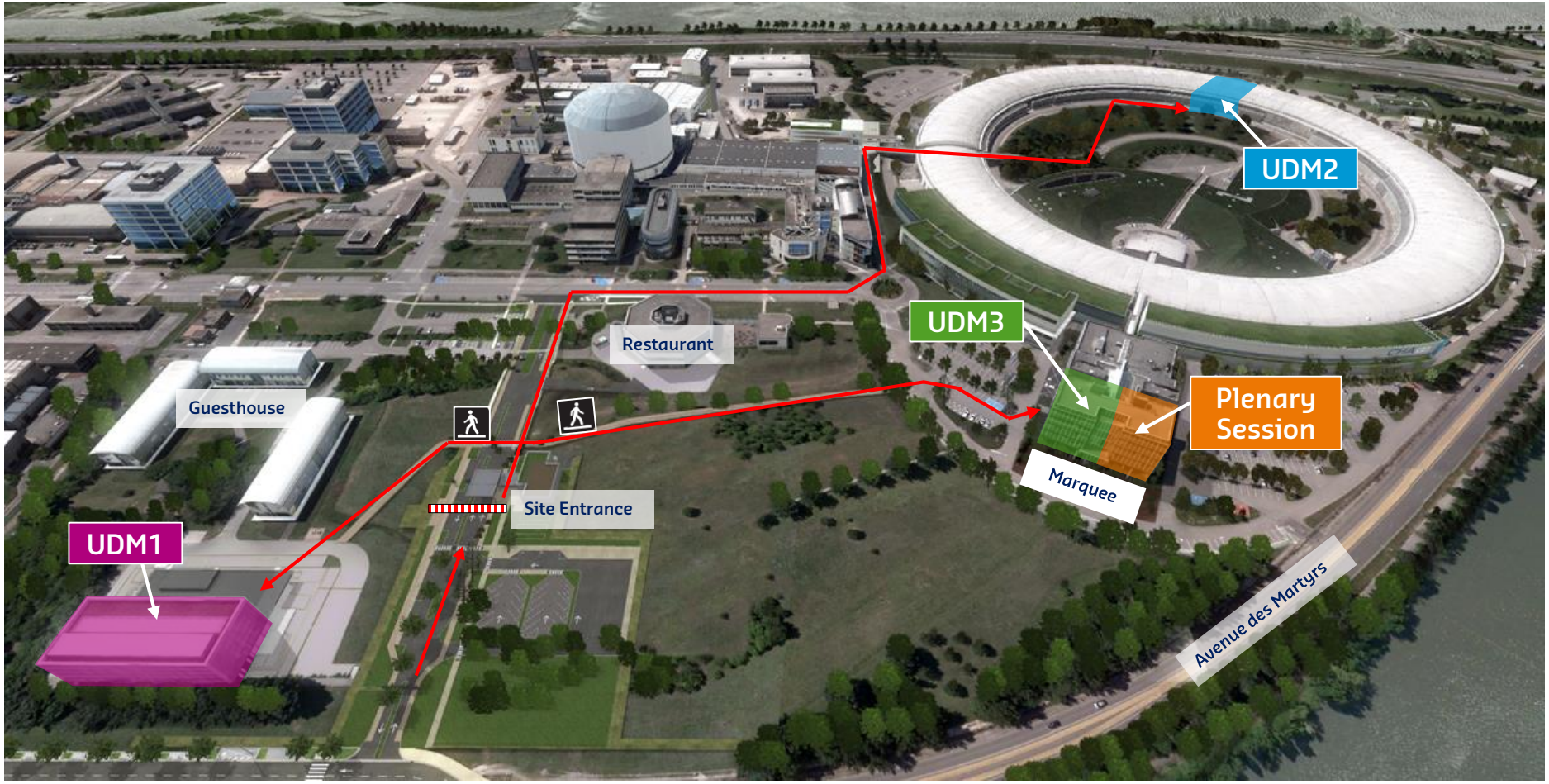
T10	Coherent imaging analysis with PyNX: CDI (small angle and Bragg), ptychography, holotomography	LOB-1-45

USER MEETING 2023 – Plenary Session & Microsymposia Venues



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Plenary Session	→	ESRF Auditorium
UDM1	Environmental sciences: challenges and opportunities under a new era of synchrotron light →	IBS Seminar Room
UDM2	Tomography at BM18 →	ESRF MD-1-21
UDM3	Operando science of functional energy conversion, storage materials and devices →	ESRF Auditorium